# A Guide

to the collection and submission of samples for laboratory analysis

AIR

**PLANTS** 

SOIL

WATER

FISH

LABORATORY

assessment enhancement research enforcement

1989



Ministry of the Environment Laboratory Services Branch 125 Resources Road P.O. Box 213 Rexdale, Ontario M9W 5L1

Attn: LCS-QA/QC Section

## QUESTIONNAIRE

THE LABORATORY SERVICES BRANCH INTENDS TO PRODUCE ANOTHER EDITION OF THIS PUBLICATION IN THE NEAR FUTURE. WE WOULD WELCOME YOUR INPUT AS A CLIENT TO ESTABLISH THE CONTENT AND FORMAT.KINDLY COMPLETE AND RETURN THIS QUESTIONNAIRE TO THE ADDRESS SHOWN ON THE REVERSE SIDE

NAME
AGENCY PHONE
SIZEBIGGERSMALLERSAME
ADD SECTION (DESCRIBE CONTENT)
MORE DETAIL (SPECIFY SECTION)
LESS DETAIL (SPECIFY SECTION)
ELIMINATE SECTION (SPECIFY)
REVISE FORMAT (SPECIFY SECTION)
WOULD YOU BE WILLING TO CONTRIBUTE TO THE NEXT EDITIONYESNO
WHAT ERRORS HAVE YOU NOTICED IN THE TEXT?
COMMENTS

## A GUIDE TO THE COLLECTION AND SUBMISSION

OF

## SAMPLES FOR LABORATORY ANALYSIS

## SIXTH EDITION

Co-ordinated by

LCS-QA/QC Section

Laboratory Services Branch

Ontario Ministry of the Environment

April, 1989

# TABLE OF CONTENTS

I.	INTRODUCTION			
	A. LA	BORATORY SERVICES BRANCH	8	
	B. SA	MPLE ACCEPTANCE POLICY	8	
	C. IN	TERLABORATORY SPLIT SAMPLE POLICY	9	
	D. OV	ZERVIEW	9	
II.	SAFET	Y CONSIDERATIONS	13	
	A. OV	VERVIEW	13	
	B. OC	CCUPATIONAL HEALTH AND SAFETY ACT 1978	14	
	C. WHMIS (Workplace Hazardous Material Information System)			
III.	SAMPL	E COLLECTION	16	
Α.	SAMPL	E COLLECTION FOR WATER QUALITY ASSESSMENT	16	
	A- I.	SAMPLE COLLECTION FOR CHEMICAL ANALYSIS	16	
		1. GENERAL CONSIDERATIONS 2. SAMPLE CONTAINERS 3. PRESERVATION TECHNIQUES 4. SAMPLE VOLUME 5. SAMPLING METHODOLOGY 6. FIELD RECORDS	16 16 17 18 18	
	A - II.	SAMPLE COLLECTION FOR MICROBIOLOGICAL TESTING	19	
		1. GENERAL CONSIDERATIONS 2. MICROBIOLOGY PARAMETERS	19 19	

	3. SAMPLE CONTAINERS 4. PRESERVATION TECHNIQUES 5. SAMPLE VOLUME 6. SAMPLING METHODS a) Surface Water Samples b) Tap Water Samples	19 20 20 20 20 21
A - III.	SAMPLING FOR ASBESTIFORM MINERAL FIBRES	21
A - IV.	SNOW-COVER SAMPLING	21
	2. SAMPLE COLLECTION	21 21 22
A - V.	GROUND-WATER SAMPLING	22
	<ol> <li>SAMPLE COLLECTION         <ul> <li>Major Inorganic Ions and Trace Element Ions</li> <li>Organic Parameters</li> <li>Dissolved Gases</li> </ul> </li> </ol>	22 22 23 23 23 23
A - VI.	CHEMICAL AND PHYSICAL FIELD ANALYSIS	23
		25 25
		25
- ***	1. DUSTFALL SAMPLING 2. HI-VOL FILTER SAMPLING 3. SAMPLING FOR ASBESTIFORM MINERAL FIBRES	25 25 26 27

В.

В -	- III. NON-ROUTINE TECHNIQUES			
	2. 3. 4. 5.	LOW VOLUME SAMPLING HIGH VOLUME SAMPLING COMBINATION GRAB SAMPLES DETECTION TUBES CASCADE IMPACTORS DICHOTOMOUS SAMPLERS STACK SAMPLING	27 27 28 28 28 28 28	
в -	IV. RO	UTINE VEGETATION AND SOIL SAMPLING	29	
	3. 4.	GENERAL TYPES OF INVESTIGATIONS a) Assessment Surveys b) Complaint Investigations SAMPLING PROCEDURES SAMPLE STABILIZATION SAMPLE IDENTIFICATION	29 29 29 29 29 30 30	
C.	SAMPLE COLLECTION FOR THE ANALYSIS OF SEDIMENT, SOLID WASTES, SOIL, AND BIOMATERIALS			
	C - I.	COLLECTION OF SOIL, SEDIMENT, SOLID WASTES, AND BIOMATERIAL SAMPLES	31	
		1. GENERAL 2. SAMPLING CONSIDERATIONS 3. SAMPLE CONTAINERS 4. PRESERVATION TECHNIQUES 5. SAMPLE SIZE 6. SAMPLING METHODS AND FIELD RECORDS	31 31 31 32 32 32	
	C - II.	APIOS SOIL SAMPLING	32	
		1. GENERAL 2. TYPES OF INVESTIGATIONS	32 33	

			SAMPLING PROCEDURES SAMPLE PREPARATION FOR SUBMISSION TO THE LABORATORY	33 33
	C - III.		GETATION SAMPLING FOR THE ACID PRECIPITATION ONTARIO STUDY (APIOS)	33
	C - IV.		LLECTION OF FISH SAMPLES FOR INORGANIC AND GANIC CONTAMINANTS ANALYSES	34
		2.	ANALYTICAL TESTING CAPABILITIES SAMPLE PREPARATION AND SUBMISSION a) Records b) Filleting c) Sample Size d) Sample Containers OTHER CONSIDERATIONS	34 34 34 34 35 35
D.	LEGAL	ANI	COMPLAINT SAMPLING	37
	D - I.	RE	SPONSIBILITIES	37
			FIELD STAFF FIELD AND LABORATORY STAFF	37 38
	D-II.	WA	TER SAMPLES FOR CHEMICAL AND MICROBIOLOGICAL ANALYSIS	38
	D - III.	SA	MPLING FOR PARTICLE IDENTIFICATION	39
			AIR SAMPLES WATER SAMPLES	39 40
	D - IV.	SAJ	MPLING FOR GAS DAMAGE COMPLAINTS	40
IV. SA	MPLE SU	вмі	SSION	41
	A - I.	GE	NERAL CONSIDERATIONS	41

A - II.	SAMPLE LABELLING AND COMMUNICATION WITH THE LABORATORY	42
A - III.	PARAMETERS AND TEST CODES	43
A-IV.	PARAMETER GROUPINGS: GROUP CODES	43
A - V.	COMPLETING SUBMISSION FORMS	43
	CODING REQUIREMENTS FOR SUBMISSION DOCUMENT     CODING REQUIREMENTS FOR REQUEST FOR ANALYSIS DOCUMENT	44 49
	HIGH PRIORITY SUBMISSION PROTOCOL .SAMPLE CONTAINER REQUISITION AND SHIPPING PROCEDURES	53 55
FIGURE I	Regions and Offices in Ontario	12
FIGURE II	Fish Filleting Diagram	36
FIGURE III	LIS Submission Form	45
FIGURE IV	LIS Request for Analysis Form	48
TABLE I	Analytical Testing Capabilities, Laboratory Services Branch	56
TABLE II	Specific Parameter Information	59
TABLE III(A)	Tests Performed by the Sediment and Soils Unit (ITC)	69
TABLE III(B)	Tests Performed by Industrial/Domestic Waste Unit (ITC)	70
TABLE IV	Sampling Supplies - Central Stores	71
TABLE V	LIS Users Committee	52
TABLE VI	Emergency Response Task Force	54

APPENDIX I	Enquiries Regarding Samples	73
APPENDIX II	Laboratory Shipping Address	81

Disclaimer: Reference in this document to any commercial product does not constitute endorsement.

#### A GUIDE TO THE COLLECTION AND SUBMISSION OF SAMPLES FOR LABORATORY ANALYSIS

## I. INTRODUCTION

#### A. LABORATORY SERVICES BRANCH

The various laboratories of the Laboratory Services Branch are equipped to perform a large number of chemical and microbiological analyses on domestic water supplies, surface waters, ground waters and domestic and industrial wastes. Fish, vegetation, soil samples, hi-vol filters, precipitation and snow samples are also analyzed by these laboratories. Special analyses can be provided for research studies or unusual pollution problems.

Decentralization of the Ontario Ministry of the Environment has resulted in the designation of six regions with boundaries as given in Figure I. Three of these, the Northwestern, Southwestern, and Southeastern, have regional laboratories located at Thunder Bay, London and Kingston, respectively. Samples collected within these regions are analyzed at the appropriate regional laboratory when analytical capability is available. When such capability is not available, these samples are analyzed at the Toronto Laboratory. Samples from selected programs within the Northeastern Region are now being submitted to the regional laboratory in Thunder Bay. The chemical tests performed by each regional laboratory as well as the Central laboratory in Toronto, are outlined in Table I. Field samplers should ensure that a laboratory is capable of analyzing the parameter(s) in question before shipping. Mobile laboratories and field stations are often provided to perform a limited number of tests in conjunction with major surveys or studies.

## B. SAMPLE ACCEPTANCE POLICY

The laboratory facilities of the Ministry of the Environment provide analytical support for the Ministry's environmental protection programs. These programs include surface and ground water quality, industrial assessment, air quality, solid-liquid waste, municipal water and wastewater, enforcement and litigation.

Samples are accepted at the laboratory for analysis if collected by Ministry of the Environment staff or other authorized federal, provincial and municipal government officials. If samples are submitted by consultants, evidence of authorization by an appropriate Ministry official must be provided. All samples must be taken and submitted using the appropriate protocols.

Samples are accepted from non-Ministry sources for other purposes such as to support programs of other government departments, provided that formalized agreements have been established with the laboratory and that appropriate sampling protocol has been employed.

Samples submitted by private individuals are not accepted unless prior authorization from an appropriate Ministry official has been obtained.

#### C. INTERLABORATORY SPLIT SAMPLE POLICY

The Laboratory Services Branch requires written notice from Ministry personnel of their requirement to submit split samples between an M.O.E. laboratory and any other laboratory.

The request must be sent to the Quality Assurance unit, Laboratory Services Branch, Rexdale, for approval prior to submission of split samples.

The Director, Laboratory Services Branch, reserves the right to refuse analysis of interlaboratory split samples when there are unresolved questions regarding the proper splitting or handling of samples, or regarding any other factors which may impact on the proper interpretation of the reported results.

The Laboratory Services Branch reserves the right to review and approve the interpretation of split sample studies. For this reason, no results for such samples will be reported until a copy of the analytical results has been received from the other laboratory.

#### D. OVERVIEW

Sample collection is the first and often the most critical stage in the step-by-step procedure used to determine the presence and level of a substance or group of substances in the environment. From the standpoint of data interpretation, it is normally assumed that a representative sample has been taken. If the sample is not, in fact, representative, then this should be noted to avoid erroneous data interpretation. Similarly, once the sample has been collected, improper use of preservation techniques to stabilize the sample or delay in transportation may lead to questionable results.

In general, the sampler's aim must be to collect a representative sample from a known position (location) and transfer it to the laboratory with a minimal change in chemical composition of the parameter of interest. It is of little value to make an accurate analysis of an incorrectly collected sample.

A computerized sample entry and data processing system (LIS) is on-line at the Toronto and Thunder Bay laboratories. All samples must be submitted to the laboratory with appropriate forms. Properly completed submission documents (see Section IV SAMPLE SUBMISSION) will minimize confusion and delays in sample processing and hence subsequent lab analyses. Judicious selection of test codes will eliminate unwarranted and superfluous analyses. A final, careful cross-check of the sample, sample label, submission sheets and test requests before shipment should eliminate most errors which result in missing or useless data. Compared to sample collection, handling, and analysis, the preparation of paperwork for sample submission takes little time but is a critical and unforgiving step.

IT CANNOT BE EMPHASIZED TOO STRONGLY THAT THE SAMPLER PLAYS A KEY ROLE IN ENSURING THAT THE DATA OBTAINED REFLECTS THE FIELD SITUATION BEING ASSESSED.

The success of an environmental sampling program depends on the application of a well-defined plan. This chapter of the sampling guide is intended to assist in formulating plans for the collection of samples. The content is biased towards laboratory needs because of space limitations. Specific sampling procedures are described elswhere (Maienthal and Becker 1976; Ontario Ministry of the Environment 1977; USEPA 1982; Environment Canada 1983). Personnel at the Laboratory Services Branch are available to help coordinate sampling and laboratory procedures (Appendix I).

The complexity of the sampling plan depends on the type of program: emergency (complaint), survey, monitoring, or experimental. Emergency or survey programs ar usually "once only" operations, whereas monitoring and experimental programs are long term. Regardless of the type of program, a few general points and references can be given. The most common questions are: "Where to sample?" and "How many samples to collect?" As a rule of thumb, minimize sampling stations (choose these carefully) and assure that sufficient samples were collected at each station to achieve the program goals. For monitoring or experimental programs, the number of samples required to achieve specified statistical confidence limits can be calculated (Bernstein and Zalinski 1983; Kratochvil 1983).

Aside from contamination by the substance of interest, the presence of other substances may lead to "matrix" problems at the analytical stage. Matrix problems may be apparent to samplers only when they are extreme. For example, if the sample is difficult and unpleasant to collect (such as some industrial wastes and leachates), it will probably be difficult and unpleasant to analyze. For complex mixtures or unusual test requests, it is best to consult the laboratory staff as early as possible. In many cases, a clear understanding of what analyses can or cannot be performed will determine the sampling protocol and sample preparation for submission. It is unsatisfactory for everyone when the final data report contains the remark "unsuitable for analysis". Equally unsatisfactory, is the failure to request appropriate tests according to the type of sample and original goals of the sampling program. In either case, the omission will not be identified until weeks after the sample was submitted. Thus, prevention is the only cure.

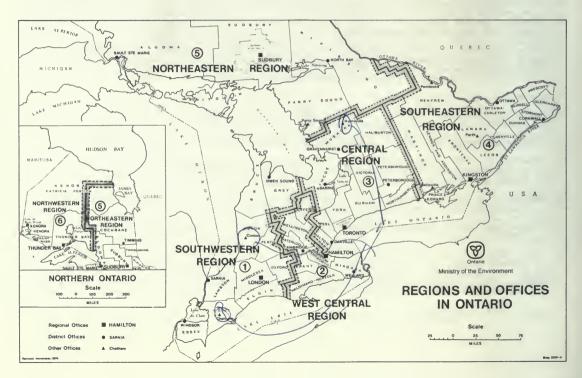
Other sampling considerations include the choice of containers, preservative and storage for samples. Tables to assist in this choice are provided. Finally, planners and samplers should consult other Ministry publications such as Outlines of Analytical Methods, 1981, for details of analytical ranges, parameters detected by method, possible interferences (matrix effects) and the average precision.

The purpose of this guide is to outline the standard procedures required to collect and submit samples to the laboratory successfully. Selection of procedure depends on the type of sample and the analytical objectives.

The sample type is fixed and will determine some of the sampling and analytical procedures. The analytical objectives are achieved by attention to specific sampling techniques, containers, sample volumes, preservative, and submission procedures. Appendix I lists contact people for the various sections of the Branch. You are strongly advised to discuss problems or large submissions with Laboratory personnel before sampling. Telephone numbers given in the guide are preceded by area code 416 unless otherwise specified. Table I provides a brief summary of tests offered by the Laboratory Services Branch (LSB). Table II provides specific information for most of these tests: container, preservation technique, minimum volume required, and comments. Tests performed by the Sediment and Soils Laboratory are described separately in Table III. Ordering information for the containers is given in Table IV. Submission procedures are given in Section IV. Table V lists the contact persons forming the LIS Users Committee. Section IV, A-VI contains the High Priority Submission Protocol, and Table VI lists the Emergency Response Task Force (ERTF) Representatives and Managers of the LSB, who must be contacted before submitting high priority samples. Appendix II gives the shipping addresses for the Central and Regional laboratories.

## References

- Bernstein, B.B. and J. Zalinski. 1983. An Optimum Sampling Design and Power Tests for Environmental Biologists. J. Environ. Management 16:35-43.
- 2. Environment Canada. 1983. Sampling for Water Quality. Water Quality Branch, Inland Waters Directorate, Ottawa.
- Kratochvil, B. 1983. Statistical Considerations in Sampling for Chemical Analysis of the Environment. In: Proc. of ACS Symposium: Role of Chemometrics in Pesticide/Environmental Residue Analytical Determinations. ACS, Seattle, WA. March, 1983.
- Maienthal, E.J. and D.A. Becker. 1976. A Survey on Current Literature on Sampling, Sample Handling, for Environmental Materials and Long Term Storage. Interface 5:49-62.
- Ontario Ministry of the Environment. 1977. Primary Treatment and Sludge Digestion Workshop. Pollution Control Branch.
- 6. Ontario Ministry of the Environment. 1981. Outlines of Analytical Methods. Laboratory Services and Applied Research Branch.
- USEPA. 1982. Handbook for Sampling and Sample Preservation in Water and Wastewater. U.S. Environ. Monitoring and Support Lab., Cincinnati, Ohio. EPA-600/4-82-029.



## II SAFETY CONSIDERATIONS

#### A. OVERVIEW

- All persons carrying out sampling activities should be familiar with the applicable sections of the Occupational Health and Safety Act 1978 and regulations for industrial establishments, in particular, those sections dealing with safeguards, confined spaces, and personal protective equipment.
- All staff carrying out sampling activities at an industrial establishment or pollution control plant should become familiar with the property, process, operation and associated hazards or be accompanied by plant personnel or another person who is aware of any potential hazards.
- 3. When sampling at manholes on streets or roadways where vehicle or pedestrian traffic may endanger the safety of any sampler, safeguards such as barriers, warning signs, traffic cones, vehicles equipped with flashing lights, fluorescent clothing, flag men or other safeguards deemed appropriate in the circumstances shall be used for the protection of all workers in the area.
- Safety footwear shall be worn when removing manhole covers or when working in any area where a worker may be exposed to the hazard of foot injury.
- 5. When sampling potentially hazardous materials such as sewage, industrial waste, or contaminated soils, the sampler shall wear the appropriate protective equipment, i.e., rubber gloves and boots, face and respiratory protection, as required. The re-entry period for pesticides shall be observed for areas which have been treated. Personnel shall observe the normal rules of basic hygiene, (i.e., wash hands and face, etc.) after sampling. Contaminated equipment and clothing shall be cleaned after each use.
- 6. There should be a minimum of two people present when sampling is potentially hazardous such as on slippery, steep, or icy creek and river banks, at street manholes, from any water craft or along any water way during hours of darkness. Work in confined spaces, such as down a manhole or wet well, requires additional precautions and equipment: gas detectors, safety harness, blower for purging, breathing apparatus, etc.
- 7. Potentially hazardous materials, (i.e., chemicals, contaminated samples) shall be protected against accidental breakage and spilling during transport. Sample bottles containing materials which may generate gases, (e.g., sludge), should not be more than half full to allow room for expansion. Potentially dangerous samples should be clearly labelled and marked "hazardous" (e.g., flammable, corrosive, toxic, explosive or radioactive) for the benefit of all persons who must subsequently handle the samples.
- Caution should be observed in handling sampling boxes and chemicals such as preservatives. Rough handling which
  may cause undue agitation to samples and other containers should be avoided. Proper safe lifting techniques should
  be employed.

9. Labelling of sample containers, boxes, etc., must be performed by the sampler in accordance with the Workplace Hazardous Materials Information System (WHMIS), an amendment to the Ontario Health and Safety Act (OHSA).

#### B. OCCUPATIONAL HEALTH AND SAFETY ACT 1978

## SECTION 16.

- 1. A supervisor shall ensure that a worker,
  - a) works in the manner and with the protective devices, measures, and procedures required by this Act and the regulations; and.
  - b) uses or wears the equipment, protective devices or clothing that his employer requires to be used or worn.
- 2. Without limiting the duty imposed by subsection 1, a supervisor shall,
  - a) advise a worker of the existence of any potential or actual danger to the health or safety of the worker of which the supervisor is aware;
  - b) where so prescribed, provide a worker with written instructions as to the measures and procedures to be taken for protection of the worker; and
  - c) take every precaution reasonable in the circumstances for the protection of a worker. 1978, c.83, s.16.

## SECTION 17.

- 1. A worker shall,
  - a) work in compliance with the provisions of this Act and the regulations;
  - b) use or wear the equipment, protective devices or clothing that his employer requires to be used or worn;
  - c) report to his employer or supervisor the absence of or defect in any equipment or protective device of which he is aware and which may endanger himself or another worker;
  - d) report to his employer or supervisor any contravention of this Act or the regulations or the existence of any hazard of which he knows; and
  - e) where so prescribed, have, at the expense of the employer, such medical examinations, tests or x-rays, at such time or times and at such place or places prescribed.

## 2. No worker shall,

- a) remove or make ineffective any protective device required by the regulations or by his employer, without
  providing an adequate temporary protective device and when the need for removing or making ineffective the
  protective device has ceased, the protective device shall be replaced immediately;
- b) use or operate any equipment, machine, device or thing of work in a manner that may endanger himself or any other worker; or

 engage in any prank, contest, feat of strength, unnecessary running or rough and boisterous conduct. 1978, c.83, s.17.

## C. WHMIS (Workplace Hazardous Material Information System)

According to WHMIS - Bill 79, amendment to the Ontario Health and Safety Act, Section 22(b) 1(a), "an employer must ensure that every container present in the workplace that contains hazardous material is and remains labelled in the prescribed manner." Consequently, if the contents of any sample are suspected to be hazardous, the container label must provide the following information:

- a) product identifier (ingredients, if known)
- b) handling instructions
- c) hazardous warning notice
- d) hazard information or source where such information can be obtained.

## III. SAMPLE COLLECTION

#### A. SAMPLE COLLECTION FOR WATER QUALITY ASSESSMENT

#### A - I SAMPLE COLLECTION FOR CHEMICAL ANALYSIS

#### 1. GENERAL CONSIDERATIONS

The method of sample collection in the field is the responsibility of the individual involved. The following points should be noted:

- a) The sample must be truly representative of the whole.
- b) All possible sources of sample contamination (sampling devices, motor exhausts, disturbing of bottom sediments, use of inappropriate containers, etc.) should be eliminated or reduced to a minimal level.
- c) Since sample composition will change with time, rapid transportation to the laboratory is desirable. For some parameters, use of a preservative is recommended. Theoretically, this should fix the concentration of the parameter of interest and reduce the need for rapid transport and analysis. However, in practice, this only delays the perishability of the parameter and the sample should still be transported as quickly as possible.
- d) For samples which do not have a preservative already in the collection bottle, rinsing both the bottle and cap with sample (two or three times) is strongly recommended. This procedure, while reducing any contamination that may be present, also tends to equilibrate the sample with the container walls and, hence, "container effects" (leaching, adsorption, etc.) are minimized. Sampling for organics is an exception since repeated rinsing may concentrate the compounds on the walls of the container.

## 2. SAMPLE CONTAINERS

Table II summarizes the proper sample container for each parameter and Table IV lists the containers available from Central Stores. Selection of containers is also determined by sample volume required (see #4 below). Special studies may require a certain container type or sample pretreatment during sample collection. It is the responsibility of the sampler to obtain the proper sample container for special studies or special analytical requests.

Sludge samples are collected in wide mouth glass or plastic bottles and never filled more than half way. The extra space is required as an expansion zone for gaseous products that may be formed. Failure to submit samples in this manner may result in container explosion during transit or at the laboratory. Overfilled sludge samples are discarded without analysis. Sludge samples for trace organic contaminant analysis should be collected in a wide mouth glass container with a foil-lined cap.

For trace level metal analysis of surface and domestic waters, PET 500 containers should be used.

Note that samples submitted for physical testing such as particle identification (see Subsection D-III) or microbiological analysis are generally unsuitable for chemical analysis and vice versa. An appropriate separate bottle should be submitted for each of these test types when more than one is required.

Most aqueous samples designated for the analysis of organic contaminants such as PCB's and pesticides should be submitted in a pretreated 1 litre brown glass bottle with a Teflon-lined cap. The container should be filled to the shoulder of the bottle. The contents should not contact anything but Teflon, glass, aluminum foil or stainless steel. Avoid contact or contamination of the samples with rubber or plastic gloves.

Aqueous samples designated for the analysis of volatile or purgeable organic constituents should be submitted in square 250 mL clear glass bottles with foil-lined caps. (NOTE: In the near future, a 40 mL glass vial fitted with a Teflon-lined septum cap will be available for this purpose.). The container should be filled to overflowing and tightly capped to exclude air.

Aqueous samples submitted for organic identification should be collected in a 1 litre clear glass bottle to allow for a visual examination of the contents. Aqueous samples submitted for petroleum hydrocarbons or solvent extractables can utilize either the clear or brown 1 litre glass bottles.

All samples collected for organic analysis should be cooled (optimal temperature  $4^{\circ}C$ , avoiding freezing) and quickly transported to the laboratory.

## 3. PRESERVATION TECHNIQUES

The function of a preservative is to stabilize the parameter of interest so that changes in composition during transit and the time prior to analysis are minimized. Several different preservation methods are recommended and these are outlined by parameter in Table 11.

Preservation techniques usually involve the addition of a chemical which "ties up" the parameter in a form which is unaffected by sample aging or else provides conditions unsuitable for any further reaction to occur. In some cases, refrigeration or freezing to reduce reaction rates provides the best preservation, particularly for those parameters which have a direct biological relationship (i.e., with respect to growth or decline, for example, nutrients).

Preservation for some parameters is effective, for others, it serves only as a technique to marginally reduce the decay or conversion rate. Therefore, it is imperative for such parameters as phenol, chlorophyll, etc., that every effort be made to submit the samples to the appropriate laboratory as quickly as possible to avoid or reduce loss.

The sampler should be aware that the use of the recommended preservative for one parameter may negate the possible analysis of another. For example, heavy metal samples preserved with nitric acid are unsuitable for nutrient analyses. It is the sampler's responsibility to determine whether use of a certain preservative will eliminate the possibility of analysis of another requested parameter, and provide suitable replicate samples to avoid the problem. If in doubt, consultation with laboratory staff is advised (Appendix 1). Each replicate should have the preservative used clearly marked on the bottle label.

CAUTION: Never add nitric acid to an empty plastic container.

#### 4. SAMPLE VOLUME

The analytical methods used to determine parameter concentrations require a certain minimum volume of sample in each case, as outlined in Table II. The field sampler is expected to calculate the total volume of sample required by summing the specified individual volumes (Table II) for all the analyses requested and to submit the appropriate quantity (see Note below). In addition, samplers are asked to submit at least 20% of sample in excess of their original total estimate, if available, to allow for possible repeat analysis. Failure to provide the required sample volume will normally result in an "insufficient sample" remark being entered on the analysis report sheets.

In most cases, the volume required for analysis depends on parameter concentration, with "clean" samples (i.e., low concentrations) needing the largest amounts. Domestic water supplies, well waters, and unpolluted surface waters fall in this category. Tests for these sample types require the largest practical volume in order to provide a sufficient quantity of the substance of interest for reliable detection. Samples of high concentration (effluents, sewages, etc.) require a much smaller amount, and even a dilution may be employed. Submission of excessive quantities of noxious, toxic, or hazardous wastes creates serious safety and disposal problems at the laboratory. If in doubt, contact laboratory personnel.

In certain cases where the sampler is unable to obtain sufficient sample volume or when resampling is impossible, analysis may still be obtained if special care and analytical techniques are used in the laboratory. This can only be achieved after consultation with laboratory personnel has been initiated by the sampler and before sample submission.

The following rules for sample volume should be followed when submitting aqueous samples for inorganic parameters (non-metals):

a) Major Ions and Nutrients (including pH, alkalinity, turbidity, DOC and COD)

- 500 mL (one PET 500 container)

b) Solids

- 500 mL (one PET 500 container) - 500 mL (one PET 500 container)

NOTE: The volume requirements for the full range of Major Ions and Nutrients (500 mL) is less than the sum of individual volumes for each parameter listed in Table II. Since certain parameter combinations are processed and/or analyzed simultaneously, the overall volume requirement is reduced.

## 5. SAMPLING METHODOLOGY

The sampler should be aware of how the particular details of his procedure (geographic location, time of day, method of obtaining the aliquot, etc.) may bias the results which are eventually obtained.

Care should always be taken to minimize sample cross-contamination by carefully rinsing (with sample) all sampling equipment used in collecting the aliquot which is sent to the laboratory. When rinsing is not possible, such as in precipitation sampling or sampling for organics, the contamination of samples by the collecting vessel or polyethylene bag should be checked regularly. These precautions are particularly important for low concentration parameters.

When sampling for volatile organics and trihalomethanes, the bottle should be tilted and filled slowly to prevent bubbling.

#### 6. FIELD RECORDS

It is in the sampler's own interest to keep complete records of his sample collection activity, not only from the standpoint of data, sample number, location, description, etc., but also with regard to unusual features which may be extremely useful in interpreting the analytical data. This information may also prove invaluable in the event of sample loss, misnumbering of sample bottles or report sheets, etc.

#### A - II SAMPLE COLLECTION FOR MICROBIOLOGICAL TESTING

#### 1. GENERAL CONSIDERATIONS

It is the responsibility of the sampler to use aseptic techniques when handling the sterile bottles used for microbiological sample collection. Failure to do so will result in sample contamination and meaningless results. It is recommended that the techniques described below be closely followed in order to obtain reliable data.

## 2. MICROBIOLOGY PARAMETERS

Table I lists the microbiological parameters performed in each laboratory with specific parameter information given in Table II. If the pollution sources are complex and/or there is doubt as to the most appropriate selection of bacterial parameters, then the Microbiology staff should be contacted.

## 3. SAMPLE CONTAINERS

Presterilized 250 mL bottles with red labels usually provide adequate volume (200 mL) for routine analyses. It is particularly important that chlorinated waters or waters in which a chlorine residual is suspected, should be sampled in presterilized 250 mL bottles containing sodium thiosulphate (red label).

Before samples are collected for sulphur cycle bacterial analyses, the Microbiology unit should be contacted to determine whether thiosulphate should be used or not.

#### 4. PRESERVATION TECHNIQUES

Sodium thiosulphate is used to neutralize the disinfecting properties of chlorine thereby preserving the existing microbial population at the time of sampling. This preservative is already present in the red labelled sample bottles. Keep samples cool, preferably through refrigeration or ice, and away from light during transportation to the laboratory. Frozen samples will not be accepted.

#### 5. SAMPLE VOLUME

In general, one bottle per sample provides sufficient volume for standard analyses. If, however, the bacterial levels expected are very low or extra parameters are being requested, then additional samples may be required. Consultation with Microbiology staff is advisable in such cases.

#### SAMPLING METHODS

Sterile sampling bottles are available through Central Stores in Toronto, and regional laboratories. For special studies, alternate bottles are obtainable through Microbiology staff on consultation. Samplers should check to see if the plastic seal on each container is intact before sampling. Containers with loose or cracked seals should not be used. All samples should be collected early in the week and shipped to the appropriate laboratory. During spring, summer and fall, samples should be packed in ice to minimize biological activity. In winter, samples should be packed in insulating material to prevent freezing while still keeping them cold. Immediate delivery to the laboratory is essential. Analysis within six hours is preferable, but should be conducted within at least twenty-four hours.

## Strict adherence to the following sampling procedures is recommended:

## a) Surface Water Samples.

Clamp the bottle onto a sampling pole before removing the cap. Touch only the outer surface of the cap when opening the bottle. The inner lip of the bottle and cap liner must not come in contact with anything except the atmosphere. If they are accidently touched, the sample has been contaminated and should not be submitted. The recommended procedure is to hold the cap with your fingertips until the sample has been taken. The cap must not be set down somewhere while the sample is being taken as this will result in contamination.

Surface sampling from a river or stream is accomplished by quickly lowering the sample bottle into the water approximately one meter below the surface with the mouth facing into the current. When sampling near shore, care should be taken to get a sample uncontaminated with sediment. When bubbles are no longer observed coming from the bottle, the bottle is then removed from the water, the water level adjusted to the top of the label, and the bottle is immediately recapped before unclamping it from the sampling pole. Samples must be collected using this prescribed technique. The use of a dipper, a sampler with a sideholder for bottles, or other sampling device will result in contamination.

## b) Tap Water Samples.

Samples from taps must be taken only after aerators, screens, hoses, etc., have been removed. Prior to sampling from a tap, the water should be allowed to run at full flow for approximately two minutes. The strong flow will clean out residual contamination around the orifice of the tap thus ensuring a more representative sample. The water pressure should then be reduced to permit taking the sample without excessive splashing which could result in contamination of the sample.

Fill the bottle to the top of the label being certain that the mouth of the bottle does not come in contact with the tap or any contaminated surface. The cap must also be handled aseptically as described previously.

#### A - III SAMPLING FOR ASBESTIFORM MINERAL FIBRES

Asbestos determination involves a time-consuming electron microscopic inspection. The extreme care and time required for this analysis make the test very costly, and very long sample back-logs are common. For these reasons, no sample should be submitted without previous consultation with the Electron Microscopy personnel.

Water samples should be collected in a PET 500 plastic bottle. Only new bottles should be used. The usual precautions of multiple bottle rinsing, rapid transport to the laboratory, etc., are of particular importance for the collection of asbestiform mineral fibre samples. Samples should be sent to the Electron Mucroscopy Unit, Inorganic Trace Contaminants Section, MOE, Resources Road, accompanied with the sample submission form.

#### A - IV SNOW-COVER SAMPLING

#### 1. GENERAL CONSIDERATIONS

A snow sampling survey should be designed to provide an adequate number of sample points to cover the area of interest. Sample sites should be in undisturbed locations, away from roads or other local sources of contamination, sufficiently open to permit the free fall of snow but not exposed to excessive drifting. Two control samples, remote from any known source of contamination are recommended for each investigation. To avoid contamination from dead vegetation or other matter near the ground, snow sampling should preferably be undertaken only when the total depth of snow exceeds 25 cm. The quantity of snow required for analysis will depend upon the types of parameters requested. Generally, sufficient snow to yield 2 L of meltwater is adequate.

## 2. SAMPLE COLLECTION

Samples are collected by means of an acrylic cylinder (6" inside diameter) which has been shown to be contamination-free for the parameters of interest (i.e., no metallic parts if metal analyses are required). The cylinder should be of sufficient length to accommodate the expected total depth of snow. Insert the cylinder into the snow to the required depth, clean the snow from around one side of the cylinder and raise the cylinder about 5

to 10 cm off the ground. Insert a hard, clean plastic plate under the base of the cylinder and remove cylinder and contents. Transfer the collected snow into clean heavy gauge polyethylene bags and retain in unmelted condition until ready for processing. Record the number of cores obtained at each site, total depth of snow, surface area sampled, the kind and amount of visible surface and subsurface contaminants. Duplicate samples may be collected at each site to avoid data loss and to assist in interpreting any anomalous results.

#### 3. SAMPLE PROCESSING

The sample material is transferred to a second clean polyethylene bag, placed in a plastic pail and allowed to melt in the bag (usually 12 - 18 hours). The volume of meltwater is measured by weighing the sample in the plastic bag (allowing for the tare weight of the bag). After vigorous mixing, to ensure uniform distribution of particulate matter, appropriate aliquots are poured into sample containers, depending upon the type of analysis and preservation treatment required. For most analyses, PET 500 containers are suitable for sample submission.

#### A - V GROUND-WATER SAMPLING

#### 1. GENERAL CONSIDERATIONS

- a) The selection of a sampling procedure should be preceded by specifying the objectives of the study and the factors which influence ground-water quality.
- b) The minimum number of parameters to achieve the objectives should be chosen.
- c) The sampler should take appropriate steps to obtain representative samples of ground-water.
- d) The analysis of certain physical and chemical parameters in the field is required. The determination of parameters such as temperature, pH, alkalinity, conductivity, and dissolved gases, etc., should be carried out in the field because changes in the sample may occur before analysis at the laboratory.
- e) For each sampling point, the sampler should keep a record of sampling conditions. For example, a description of well location, depth, construction static level, number of well volumes evacuated, details of sampling method and equipment and any other relevant points.

## 2. SAMPLE COLLECTION

Most of the comments in Subsection A-I regarding sample collection for chemical analysis are applicable to ground-water samples. The procedure for sample collection and treatment for specific analyses follow.

## a) Major Inorganic and Trace Element Ions.

Some studies may require the samples to be filtered in the field. For inorganic parameters, filtration through a 0.45  $\mu m$  membrane filter is desirable before preservation and/or analysis to exclude any undissolved (suspended) material present at the sample source. If field filtration is not possible, refrigerate and filter sample within 24 hours. Following filtration, samples to be analyzed for major inorganic anions should be preserved by refrigeration. Samples for cation analysis should be placed in PET 500 containers, and preserved with HNO3 (Table II).

The filtration of clean ground-water samples may not be necessary. However, these samples should be preserved as indicated above.

## b) Organic Parameters.

Field filtration must not be employed for sample contents to be analyzed for organic parameters.

## c) Dissolved Gases.

Ground-waters may contain  $CO_2$ ,  $O_2$  and  $N_2$ ,  $CH_4$  and  $H_2S$  which are derived from biochemical processes, and organic gases resulting from contamination.

Where a field determination of the dissolved gases is impractical, special sampling and storage techniques are required. Samples should be collected in hard glass, chemically resistant bottles. The bottle is filled to the top with no air space remaining beneath the cap. The bottled sample should be kept at a temperature slightly lower than that at which the water was collected.

To sample gases released from ground-water samples, water is displaced from an inverted calibrated glass tube which is submerged in a larger container which receives water from a pumped discharge line at a known flow rate. The glass tube is sealed under water with a special cap containing a septum, and the sample is preserved by refrigeration.

## 3. RECOMMENDATION

Investigators are advised to discuss the objective of each study with the Regional Hydrologist. The Hydrologist can assist in designing the sampling program and suggest practical procedures for the collection of ground-water samples.

## A - VI. CHEMICAL AND PHYSICAL FIELD ANALYSIS

The perishability of some parameters for which no chemical preservative is suitable necessitates field measurement. In the case of major field studies, a field laboratory facility for this purpose may be warranted. For example, such

parameters as dissolved oxygen, dissolved carbon dioxide, free chlorine, chloramines, hydrogen sulphide and temperature are extremely perishable so on-site analysis is recommended. Temperature and dissolved oxygen are conveniently measured using electrode sensors (and/or Winkler titration for dissolved oxygen) while dissolved carbon dioxide, free chlorine, chloramines, and hydrogen sulphide require more complex analytical techniques. Prior consultation with the Laboratory Services Branch, Water Quality staff, is recommended in these cases.

## B. SAMPLE COLLECTION FOR AIR QUALITY ASSESSMENT

#### B - I GENERAL CONSIDERATIONS

The reliability of final results reflects the care and procedure used to collect samples. The sampler should ensure that samples collected for air quality assessment are representative of the whole and that all possible sources of sample contamination are either eliminated or minimized. Since in some cases, specialized routine and non-routine techniques are involved in sample collection, the sampler is advised to consult laboratory personnel (Appendix I) prior to initiating a survey. Information provided should include sampling locations, frequency, analytical requirements, etc. A written outline of the sample survey should also be provided. Refer to the specific sampling technique below for information regarding the best way of transporting samples to the laboratory.

Testing capabilities are outlined in Table I, with specific parameter information given in Table II.

## B - II ROUTINE TECHNIQUES

#### 1 DUSTFALL SAMPLING

A clean sealed polyethylene dustfall collector jar (30 cm tall x 15 cm diameter) containing a polyethylene insert, identified by station number, is attached to a suitable supporting bracket, uncovered, and allowed to collect settleable particulate matter over a one-month period. Collectors are located to provide dustfall samples that are representative of the area being studied.

After a 30-day exposure period, the polyethylene insert is removed, heat sealed and transported to the laboratory in partitioned boxes or coolers.

It is very important that a record of station number and installation and removal dates accompany the collector since this information is necessary to calculate the results.

## 2. HI-VOL FILTER SAMPLING

The collection of suspended particulate matter involves filtration of air through a 20 cm x 25 cm (8" x 10") glass fibre filter using a vacuum pump capable of drawing at least 1.3 m³/minute. The normal sampling period is 24 hours. A complete description of the Hi-Vol sampling device and procedure may be obtained from the ASTM publication, Gaseous Fuels; Coal and Coke; Atmospheric Analysis, Part 26, November, 1980.

Pre-weighed and coded glass fibre filters and protective envelopes are available from the Air Quality Unit, Inorganic Trace Contaminants Section, for use with these samplers. The filter must be carefully installed (rough side upwards) on the sampler, and the coded number recorded on the envelope. Ripped or punctured filters must be discarded.

After carefully removing the filter, fold it in half along the 20 cm width, particulate side inwards and place in the corresponding envelope. Any comments peculiar to the sampling conditions should be noted. This is important for data evaluation.

The filter should be mailed to the Air Quality Unit, Inorganic Trace Contaminants Section, along with the calculated air volume. Hi-Vol filters for the Northwestern Region are obtained from and sent to the Regional Laboratory in Thunder Bay.

Certain tests for inorganics are incompatible with the glass fibre filters normally used. These include: Al, Ba, B, Ca, Na, K, Si and F. For these elements, other filter types are recommended and are available from the ITC Section for special surveys.

The filter envelope must contain the following information:

- a) Station number (i.e., sampling location)
- b) Hi-Vol instrument number, date and time of exposure
- c) Filter number
- d) Operator
- e) Flow readings at start-up and shut-down
- f) Comments regarding incidents peculiar to the sampling period
- g) Air volume (m3).

## 3. SAMPLING FOR ASBESTIFORM MINERAL FIBRES

The analytical technique for the determination of asbestiform fibres in air involves a time-consuming electron microscopic examination of the processed samples. The expertise, time and instrumentation required for this analysis make the test very costly. For these reasons, sampler discretion regarding submission of samples is required. Every attempt should be made to preserve the integrity of the sample.

Asbestiform minerals as suspended air particulates are collected on a 0.4 µm pore size Nuclepore filter using a modified Hi-Vol sampler. The modification consists of installing a flange with a 2 cm diameter opening on the air exit of the sampler. This opening acts as a limiting orifice and brings the air flow rate into a suitable measurement range. It is recommended that the Hi-Vol sampler be equipped with a transducer and an air flow rate recorder. The sampler must be recalibrated after the modifications have been performed. Procedures for calibration may be obtained from the laboratory or from the Technical Support Group, Central Region, telephone 424-3000.

It is very difficult to change the filter in the field and pre-installation of the filter in the Hi-Vol cassette inside an enclosed area is recommended. The entire cassette assembly is then attached to the air sampler. Removal of the filter should be performed in the reverse order.

After exposure, the filter is removed from the cassette, placed on the 20 x 25 cm separator sheet supplied with the filter, and both are then folded along the 20 cm width. The folded filter and separator are placed within a glassine

envelope and mailed to the laboratory in the usual kraft paper Hi-Vol envelope, together with all pertinent sampling data. Samples requiring asbestos analysis should be sent to the Electron Microscopy Unit.

#### 4. FLUORIDATION AND SULPHATION RATES

Fluoridation rates are measured using the appropriate candles or plates, while sulphation rates are measured using the appropriate plate. These devices may be obtained from the lnorganic Trace Contaminants Section. Protective shelters are provided and installed by the regional staff. Normal exposure time is 30 days. The exposed candle or plate should be carefully replaced in its protective cover, placed in its shipping container and sent to the LSB with the appropriate LIS form. Proper sealing of the candle or plate is important to prevent further atmospheric reaction occurring during transit. The duration of exposure must be recorded and submitted with the candle or plate. A complete description of sampling considerations for sulphation rate can be found in the ASTM Gaseous Fuels; Coal and Coke; Atmospheric Analysis, Part 26, November, 1980. Plates are recommended for any new surveys.

## B - III NON-ROUTINE TECHNIQUES

The following techniques may be used in special circumstances after discussion with laboratory staff. The sampling of volatile organic contaminants are covered by some of these techniques.

#### 1 LOW VOLUME SAMPLING

"Low volume" techniques include sampling with impingers, adsorption tubes, and filters. Samples for the analysis of volatile organic components such as vinyl chloride, peroxy-acetyl-nitrate (PAN), volatile aliphatic and aromatic hydrocarbons, and volatile organohalides may be collected by passing 100 to 1000 mL of air per minute through a specially prepared tube containing Carbosieve and Carbotrap B, or another suitable adsorbent. The normal sampling period is 2-4 hours. The tubes are available from the Trace Organics Section. The sample, once collected, must be refrigerated and kept in the dark. The sample label attached to the tube must have the following information marked on it:

- a) Date and location
- b) Pump time on and off
- c) Air flow rate at the start and finish
- d) Wind speed, direction, and temperature.

Samples should be sent to the Trace Organics Section.

#### 2. HIGH VOLUME SAMPLING COMBINATION

"High volume" techniques use a combination of Teflon-coated glass fibre filters and an XAD-2 resin cartridge. Samples for the analysis of semi-volatile compounds such as chlorinated benzenes, polychlorinated biphenyls

(PCB's), poly-aromatic hydrocarbons (PAH), organochlorine pesticides may be collected by passing 700 to 2800 cubic meters of air through the sampling device over a 24 to 96-hour period. Sampling devices may be obtained from the Trace Organics Section. Special sampling equipment must be available for sample collection.

#### 3. GRAB SAMPLES

An alternative way of sampling for volatile contaminants is by collecting a "grab" sample in Tedlar bags, aluminized Mylar bags, evacuated glass and metal containers, etc. A grab sample is taken by pumping air into the bag or filling an evacuated container with air. This sampling method may be applicable in cases of odour problems, specifically volatile organic and inorganic sulphurous compounds such as H<sub>2</sub>S and mercaptans.

An important consideration is that the contaminant does not react with or adsorb on the material of the container.

#### 4. DETECTION TUBES

By observing the length of discolouration produced in a solid absorbent of a specific tube through which a known small volume of air is drawn, the approximate concentration of a pollutant can be estimated. This method is a rapid, semi-quantitative procedure for measuring high levels of gaseous pollutants (SO<sub>2</sub>, CO, H<sub>2</sub>S) in the field.

#### 5. CASCADE IMPACTORS

Impactor type samplers capable of separating particulate matter in to size ranges according to their aerodynamic size are available. The cascade impactor separates particulate matter within the respirable range  $(0.3 - 10 \ \mu m)$ .

The sampler may be used for differentiating between sources of pollution. For example, lead emitted from automotive sources is found in the sub micrometer fraction, while lead emitted from certain industrial operations as particles is depositied in the larger than  $1 \mu m$  fractions.

The impactors are used in association with Hi-Vol samplers. A problem specific to the cascade impactor is that the jets become clogged with dirt and require frequent cleaning to maintain its calibrated flow rate.

## 6. DICHOTOMOUS SAMPLER

Recently, a new type of air fractionating instrument, called a dichotomous sampler, has come on the market. It separates the dust into two size fractions, less than 2.5  $\mu m$  and 2.5 - 10  $\mu m$ . The samples are collected on inert filters, which are ideal for rapid chemical analysis using x-ray fluorescence.

## 7. STACK SAMPLING

Stack samples can be obtained by inserting probes into a vent through which gaseous or particulate emissions pass to the atmosphere. Analytes such as chlorinated benzene, chlorinated phenols, polychlorinated biphenyls (PCB's),

polyaromatic hydrocarbons and volatile organic compounds may be collected with specially designed sampling equipment. Emission rates can be calculated from analysis of samples. Rigid procedures must be followed in stack sampling to ensure representative samples are taken. Most of this sampling is carried out by experienced outside agencies. Analytical work on stack samples has been carried out in conjunction with investigations on special industrial source emissions. Special arrangements must be made with laboratory personnel prior to sample collection.

#### B - IV ROUTINE VEGETATION AND SOIL SAMPLING

#### 1. GENERAL

The Phytotoxicology Section, Air Resources Branch (880 Bay Street), is responsible for the investigation of all complaints concerning suspected air pollution damage to vegetation or contamination of soil, and the establishment of all vegetation and soil assessment surveys in the vicinity of proposed or existing industrial emission sources. The exception is in the Northeastern and Northwestern Regions, where the work is performed by the Technical Support Sections, with assistance as required from Phytotoxicology personnel. A complete field investigation procedural manual has been prepared for use by trained personnel from these sections.

#### 2. TYPES OF INVESTIGATIONS

## a) Assessment Surveys.

These surveys are conducted to document endemic conditions prior to the establishment of emission sources, to define the current state of air emissions from existing sources, and/or to monitor source compliance with Ministerial orders. Normally, a sampling grid is constructed, centered on the source and samples are taken from established stations, located at increasing distance along radii from the source to the limits of suspected contamination. Consideration is given to the location of air quality monitoring instruments and meteorological parameters such as prevailing wind direction.

## b) Complaint Investigations.

Samples may also be taken to evaluate situations where extensive damage to vegetation has been observed. Cases of this nature will usually be drawn to the Ministry's attention through complaints by individual citizens. All complaints of this nature should be referred to the Phytotoxicology Section. They will be investigated and reported to the Regional Manager for distribution to the individual originating the complaint and to the source of the contaminant.

## 3. SAMPLING PROCEDURES

To ensure a correct interpretation of analytical data, all samples that are to be compared must be carefully matched with regard to plant species, age or maturity of leaf tissues, age of tree or shrub, and position of

sample on tree or shrub. Usually, foliage is collected from the side of the tree or shrub facing the presumed source of air pollution but, occasionally, a second sample may be taken from the side opposite from the source. Samples are taken by trimming outside growth from ground level up to 6 meters or more and collecting all leaves to provide a composite sample of 500 to 1000 grams of fresh material.

Samples are placed into perforated polyethylene bags and are transferred to refrigerated storage as soon as possible for processing in the Phytotoxicology laboratory. Forage samples (grass) are collected by cutting the terminal 25 cm of stems and blades over the representative area to be sampled. Dried flower heads and stalks are discarded and no root material whatsoever is included. The different forage species included in the sample are identified and should be representative of the population of the species in the field.

Any sample contaminated by roadside dust should be noted in the accompanying request form.

Soil samples are normally collected in conjunction with vegetation samples as an aid to differentiate between current and past emission situations. Occasionally, soil samples will be collected to establish background conditions.

Soil is collected with a 2 cm diameter stainless steel tube. A minimum of 10 cores is taken from a representative area of the sampling site. The collection form is completed to comprehensively describe the texture of the soil and the overall sampling site. Depending on the survey emphasis, the cores may be separated into fractional depths of 0-5 cm, 5-10 cm and 10-15 cm. Each level is placed in an appropriately labelled plastic bag.

Ideally, soil should be sampled from an undisturbed or sodded area and contaminated situations should be as closely matched as possible with conditions existing immediately ouside of the area.

## 4. SAMPLE STABILIZATION

All vegetation samples, as collected, are potentially unstable, and will decompose unless properly handled. Care is taken to ensure that samples are not exposed to the sun and are placed in refrigerated storage until they can be processed. When dried at 80°C for 36 hours in a forced draft oven, they become almost permanently stable.

Soil samples are spread out on non-metallic trays and air-dried for a minimum of 48 hours, or until completely dry.

## 5. SAMPLE IDENTIFICATION

Collection of vegetation and soil samples is accompanied by the use of pre-numbered identification stickers and the completion of special Terrestrial Effects LIS forms which will later provide all the necessary information required for interpretation of the test results. A portion of the numbered stickers is detachable and is placed in with the sample for identification. Normally, samples are "double-bagged" with the numbered stickers placed in the inner bags.

## C SAMPLE COLLECTION FOR THE ANALYSIS OF SEDIMENT, SOLID WASTES, SOIL AND BIOMATERIALS

## C - 1 COLLECTION OF SOIL, SEDIMENT, SOLID WASTES AND BIOMATERIAL SAMPLES

#### GENERAL

The Inorganic Trace Contaminants Section - Soils and Sediment Unit performs sample digestion or leaching on solid samples for a number of tests performed by other sections, as well as performing unique sediment analyses. These are given in Table III along with the required sample size and method of preparation.

The Trace Organics Section performs a number of analyses on soils and sediment samples such as PCB's, chlorophenols, and petroleum hydrocarbons. Glass containers must be used for samples requiring organic analysis. For further information, contact the Trace Organics Section.

#### 2. SAMPLING CONSIDERATIONS

- a) Where possible, composite sampling will result in a more representative sample than a single grab.
- b) All possible sources of sample contamination should be reduced to a minimal level.
- c) Chemical preservatives are generally not applicable to samples of this type.
- d) Preparation of these types of samples for chemical analysis generally takes longer than for water or effluent. As a result, samples requiring immediate attention should be so marked and the laboratory should be notified well in advance. Notification of a heavy sample and/or test input is essential.
- e) Samplers should be aware of pertinent information regarding submission procedures as outlined in Section IV.

## 3. SAMPLE CONTAINERS

Any clean glass or plastic container is acceptable for sediment, soils or biomaterial samples. While not recommended, paper bags can be used for "dry" soil samples where contamination from the container is not anticipated to affect the analysis (e.g., particle size analysis). In general, wide mouth 60, 125, 250, 500 or 1000 mL glass or plastic containers are the most suitable, depending upon sample size. When a number of samples are submitted as a series, uniformity of sample container size is recommended for shipping, handling and storage convenience. Plastic bags (Whirl-pak type) are usually adequate for dried vegetation samples. Containers should be clearly labelled and samples numbered in sequence, preferably from No. I. Indicate the number of containers for a sample when there is more than one.

## 4. PRESERVATION TECHNIQUES

Chemical preservation techniques are generally not applicable to solid type samples. In the short term, storage at  $4^{\circ}$ C or freezing will minimize the transformation of species, particularly if a soluble or "available" parameter is desired.

Drying as a preservation technique is recommended except for those samples requiring analysis of potentially volatile parameters. Information on appropriate drying procedures can be obtained from the ITC Section. In general, dried samples are indefinitely stable.

Air or oven drying of vegetation samples at 80°C and grinding with a Wiley mill before submission to the laboratory is recommended and where this is not possible, the laboratory must be notified so that the samples can be dried without delay. This is particularly important since the nutrient content of plant material could be significantly altered by decay.

#### SAMPLE SIZE

The field sampling personnel must be aware of the general non-homogeneity of solid samples and thus the minimum sample size should reflect this consideration. As a general rule, however, a sample which will yield 10-25 g of dry material will be sufficient for all the routine chemical analyses. Special analyses will require more sample, depending upon the tests requested. Where it is not possible to obtain a sufficiently large sample, special arrangements can be made with the laboratory personnel to perform the analyses in a given sequence so that the more important tests will be completed first.

If leachate potential tests are required on industrial waste materials, larger sample sizes will usually be required (500 g). Do not dry before shipment.

## 6. SAMPLING METHODS AND FIELD RECORDS

The sampling personnel are usually in control of the sampling methodology and must be aware of how the particular details of the procedures and sampling apparatus (e.g., coring versus dredge devices) may bias the results which are eventually obtained.

## C - II APIOS SOIL SAMPLING

## 1. GENERAL

Soil samples connected with the APIOS program are analyzed in the Dorset soils laboratory. Analytical support is provided for APIOS soil studies conducted by Air Resources Branch (Task 3) and Water Resources Branch (Tasks 2 and 4). A unique set of soil tests was developed specifically for the APIOS program and if non-APIOS samples are to be sent to this lab for analysis, arrangements must be first made with A. Neary in Dorset (705) 766-2412.

#### 2. TYPES OF INVESTIGATIONS

Soil sampling is currently done as part of the Soil Baseline Study, Forest Productivity and Decline studies and the Biogeochemistry study.

#### 3. SAMPLING PROCEDURES

For most purposes, soil samples are taken by horizon from the face of a soil pit. A pit of approximately 1 m² is dug to a depth sufficient to permit sampling of the parent material. Soil profiles are described according to criteria adopted by the "Canadian System of Soil Classification" (1978). The soil pit is photographed and a sketch is drawn of one of the faces sampled to show major soil horizon boundaries and the position of stones, root masses, and sampling points.

Beginning with the deepest horizon, and while wearing plastic gloves, samples (approximately 1 kg) are collected with a hand trowel and placed in labelled plastic bags. Duplicate samples, either from opposite faces of the pit or side-by-side from the same face, may be taken. After sampling, soil horizons are returned to the pit in the reverse order from which they were removed to minimize disturbance of the sampling site.

For some studies, it may be preferable to use a soil corer for analysis. If so, the horizons should be separated and the depth of the samples noted. Collect samples in labelled plastic bags.

#### 4. SAMPLE PREPARATION FOR SUBMISSION TO THE LABORATORY

Within a few days of collection, samples should be spread out on non-metallic trays and air-dried for a minimum of 48 hours. Samples are then disaggregated with a mortar and pestle and sieved to <2 mm (10 mesh ASTM). This fraction is submitted to the lab in a labelled glass jar. Polystyrene is unacceptable as the fine colloidal particles tend to stick to the sides of the jar, biasing the sample. A subsample of the <2 mm fraction is then ground in an agate mortar and pestle until the whole subsample passes through a 500 µm (35 mesh ASTM) sieve. This ground sample is only required if the following tests are requested: N, P, C, S, pyrophosphate, dithionite or oxalate extractable Fe, Al or Mn, or heavy metals. The ground sample should be submitted in a glass vial (Wheaton scintillation vials, 30 mL, recommended). Test groups TEAPS1, 3 and 6 are used for APIOS soil samples.

### C - III VEGETATION SAMPLING FOR THE ACID PRECIPITATION IN ONTARIO STUDY (APIOS)

The bulk of the vegetation samples connected with the APIOS program are collected in accordance with procedures established by the Air Resources Branch for the Forest Productivity and Decline study. For details, contact D. McLaughlin, Air Resources Branch, (416) 965-4516. In 1989, a report will be available outlining standard sampling methodologies for vegetation and soils samples connected with LRTAP monitoring programs.

Once collected, samples are processed as for routine vegetation samples (see Section B-IV).

#### C - IV COLLECTION OF FISH SAMPLES FOR INORGANIC AND ORGANIC CONTAMINANTS ANALYSES

#### 1. ANALYTICAL TESTING CAPABILITIES

Fish samples, normally muscle tissue, are frequently analyzed for mercury or other metals by the Inorganic Trace Contaminants Section; pesticides, PCB's, and other organics by the Trace Organics Section, Dioxin/Furans by the Drinking Water Organics Section. The following sample procedures should be closely followed in order to obtain meaningful data as to the existence and degree of contamination.

#### 2. SAMPLE PREPARATION AND SUBMISSION

#### a) Records.

Details of catch, total length (cm), weight (g), sex (if possible) must be neatly recorded on the submission sheet. For proper statistical evaluation, there should be a minimum of 10 fish per species. As with all samples, the recuired analyses should also be indicated on the submission sheet.

### b) Filleting.

Samplers are requested to submit fillets (rather than the whole fish) for analysis. Normally, the analysis is carried out on tissue from the epaxial muscle (Figure II) by making an incision with a stainless steel knife on the dorsal surface of the fish as shown (Incision No. 1). The epaxial muscle is then removed by cutting from the initial incision toward the tail (Incision No. 2) until a sufficient quantity of tissue is obtained. The muscle may be finally separated from the body (Incision No. 3). The skin should be removed from the sample.

It is important not to remove tissue from below the lateral line because of the high fat content in this region which makes PCB analysis impractical. The sample should be frozen immediately after filleting and transported to the laboratory in this condition. This is the only acceptable preservation technique. When a collection is ready for shipment to the laboratory, please phone prior to sending. For mercury or metals, contact Darryl Russell at 235-5857. For PCB's or pesticides, contact Dan Toner at 235-5755.

## c) Sample Size.

The minimum and preferred quantities of tissue required for each type of analysis are as follows:

	Absolute Minimum (g)	Preferred (g)
Mercury	20	50
Other Metals	50	100
PCB, pesticides	10	100
Dioxin/Furans	50	500

### d) Sample Containers.

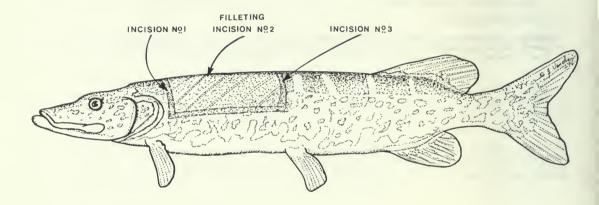
Individual samples collected only for metals and mercury analysis may be placed in small plastic bags and then frozen. Clear identification with a sample code using a masking tape label is recommended, while the use of some variety of water-proof ink is a necessity.

Samples collected for PCB or pesticide analysis must be wrapped in solvent-washed aluminum foil prior to freezing. Multiple washing of the foil and knife with hexane or acetone is a necessity. Samples submitted in plastic bags for these analyses will not be accepted. When both mercury and PCB's are required, submit the sample (frozen) in solvent washed foil.

#### 3. OTHER CONSIDERATIONS

Analysis of tissues other than muscle is possible but can only be done by special arrangements. Any further queries should be directed to Darryl Russell (235-5857), Inorganic Trace Contaminants Section, or Dan Toner, Trace Organics Section, (235-5755).

FIGURE 2



#### D. LEGAL AND COMPLAINT SAMPLING

Sampling in connection with legal action naturally requires special care due to the influence this sampling may have on case outcome. Court cases are usually initiated to determine legal responsibility for reported pollution events (stream, well contamination, vegetation or paint damage, etc.) and sampling must be conducted with this purpose in mind. In general, standard sampling methods as described previously may be used; however, the following additional points and techniques should be fully read and understood before taking any legal samples.

#### D - I RESPONSIBILITIES

#### 1. FIELD STAFF

- 1. Samples should be designated as "Legal" only when results may be used for prosecution or appeal proceedings.
- 2. Samples should be collected and transported to the Laboratory according to recommended procedures or according to specific instructions from appropriate Laboratory personnel.
- Submission of Legal samples should be preceded by a phone call to the Legal Sample Co-ordinators or Alternates (235-5863).
- All submissions should be accompanied by a full description of the problem and sampling details. Reference to previous related samples should be made, if possible.
- 5. All samples must be submitted through the Laboratory Information System (LIS) with Submission and Request for Analysis forms completed and included with the samples or forwarded to the laboratory as soon as possible. The Case Submission Receiving Report should also be filled out and enclosed with the submission forms.
- Descriptions, sample locations, times, and dates given on the Request for Analysis forms must match those given on sample container labels. Any changes must be accounted for and documented by the field staff submitting the samples.
- 7. All samples listed on the Request for Analysis forms must be accounted for.
- 8. All sample containers or packing boxes must bear seals, preferably Ministry of the Environment, or locks. If paper seals are used, the seal tags should be affixed to the Request for Analysis form beside the corresponding sample.
- 9. The chain of custody must be maintained and documented. If this is not documented with the submission, the sampler will be contacted to determine whether the submission is still viable for litigation. Copies of shipping way bills must be maintained.

- 10. All samples known to contain or suspected of containing hazardous materials MUST be labelled as such.
- NOTE: Field personnel are responsible for the submission until it is received and signed for at the Laboratory by a Provincial Analyst/Officer.

NOTE: Field staff are responsible for tracking down samples lost by third party carriers.

#### 2. FIELD AND LABORATORY STAFF

- 1. The investigating officer(s) should be in contact with the appropriate Provincial Analysts well in advance of appearing in court to discuss and clarify any interpretation of results, statements of environmental impact or recommendations which have been made in those Provincial Analysts' Certificates of Analysis or which are likely to be made in Court.
- All requests for Provincial Analysts to appear in court or at meetings to discuss Legal submissions, should be made through or passed on to the Legal Sample Co-ordinators.
- 3. Field and Legal staff are responsible for detailing what materials Provincial Analysts should have available for the Court. These materials may include sample containers, raw results, log books, etc.
- Provincial Analysts are responsible for having any materials specified by Field and Legal staff available for the Court.
- All Provincial Analysts will have an up-to-date resume on file with the Legal Samples Co-ordinators. A listing
  of published papers should also be provided. A copy of each Provincial Analysts' resume will be sent to Legal
  Services.

## D - II WATER SAMPLES FOR CHEMICAL AND MICROBIOLOGICAL ANALYSIS

The following points should be precisely adhered to when collecting court case water samples requiring chemical and microbiological analyses:

- a. The sampling area should be completely "walked", i.e., checked over at the time samples are taken, so that the sampler is completely familiar with the overall geographic "picture". The sampler must identify ALL possible contamination sources, unusual occurrences, and a "blank" sample location far enough away (upstream) that no contamination from the sources in question can influence it. The sampler should also prepare a sketch map of the area.
- b. The sampler should be careful to obtain samples at all possible contamination sources, not just the one in question.

  The observed contamination should be traced back to its source, and samples collected at key points to show continuity. In the case of an underground sewer, when the defendant or official agent is unwilling to confirm

continuity of flow of wastes through the sewer, in front of a witness, the sampler should verify continuity by passing some small, identifiable floating object through the sewer and recovering it at the outfall. Similarly, a series of samples downstream is advised to show how the contamination effect persists. A prerequisite is a "blank" unaffected by the alleged pollution (obtained upstream, or from a nearby well, etc.).

- c. The sampler should obtain prior knowledge of exactly what type of contamination he is dealing with (i.e., what parameters(s) will be measured), and sample accordingly with respect to correct bottles, preservative, etc.
- d. Legal samples must be analyzed in duplicate and thus it is recommended that at least three times the normal sample volume be submitted. Any remaining sample may then be used for further confirmation or presentation in court. All legal samples taken for microbiological analyses must arrive at the laboratory within 24 hours after sampling.
- e. It is preferable, but not essential, that the actual sampling be performed with the assistance of a witness who is willing to sign an affidavit and appear in court, if necessary.
- f. A complete and accurate record of sampling locations, time and date, bottle numbers, preservatives, etc., must be made. Submission sheets should accompany the samples in the normal manner. However, it is emphasized that the sample description and number of the bottle must exactly correspond to that on the sheet. If not, the Certificate of Analysis can be questioned, and may not be accepted as evidence.

#### D - III SAMPLING FOR PARTICLE IDENTIFICATION

In many instances, generally arising from citizens' complaints, it becomes necessary for field personnel to collect samples for constituent identification by means of microscopic, x-ray diffraction, electron probe and other techniques. The types of material normally encountered are visible solids present in air or water, that are a cause of nuisance or concern to the complainant. General guidelines to be used for sampling are given below.

#### AIR SAMPLES

Dust fallout is a most frequent cause of complaints. Dust, adhering to any surface, may be removed by lifting it by means of transparent tape. While sampling with the tape, it is useful to the analyst if the damaged spots or particles are circled on the nonsticky side of the tape with a pencil or pen. Whenever tape has been used for sampling, it should be protected by means of covering strip which comes with the tape or attached to a glass microscope slide. UNDER NO CONDITIONS SHOULD THE TAPE BE FOLDED ON ITSELF. When sampling suspected soot fallout (especially oil soot), the use of tape is not advisable, as the pressure used in collecting it often destroys the identifying characteristics. In such cases, it is better to remove a small paint section from outside window sills, shutters, etc. Plant leaves, eavestroughing, bird baths, furnace and air conditioner filters often act as collectors of particulate fallout. Where the fallout occurs consistently, aluminum weighing dishes, wetted with a glycerin-water mixture, can be used as miniature dustfall jars. These can be attached to a suitable vertical surface by means of a thumb tack. Samples can also be collected using a household vacuum cleaner and a millipore field monitor, by means of a simple adapter placed between the monitor and the vacuum cleaner hose.

As a general rule, samples should not be collected from non-stationary objects such as automobiles, since the source of the dust may then be in question. Damage to automobile paint or house sidings is generally caused by very acidic or basic materials attacking the paint surface. Such types of fallout should be tested on the spot using pH indicator paper. It is often difficult to remove a representative sample from such surfaces, and on-site inspection by laboratory staff may be necessary to determine the cause of the damage.

Heavy dustfall onto snow should be sampled by scooping the snow into a large-mouth glass or plastic bottle in such a way as to maximize the amount of particulate material obtained and prevent any possible contamination from underlying soil.

All samples collected as a result of air pollution complaints should be accompanied by the Analytical Request and Inspection report forms, which should provide all the information required to make a proper assessment of the situation.

A sketch map of the area is strongly recommended. Comparison samples of the suspected contaminants are always very useful in obtaining a positive identification of the fallout. Forward samples and forms to Rusty Moody, Inorganic Trace Contaminants Section, Resources Road, (235-5863).

#### 2. WATER SAMPLES

Water samples that require identification of suspended solids may be sent to the laboratory in any of the standard containers. A few milligrams of material are usually sufficient for microscopic analysis, although for complex mixtures requiring multi-instrumental analyses, 2-3 g of material would be preferable. Submit samples to Rusty Moody, Inorganic Trace Contamininants Section, (235-5863) if the material appears to be principally inorganic; if organic, submit to J. Osborne, Trace Organics Section (235-5759). If the material appears to be biological, submit to G. Hopkins, Aquatic Biology Section (235-5811).

#### D - IV. SAMPLING FOR GAS DAMAGE COMPLAINTS

In cases of suspected gas damage, the stained surface, such as paint work, should be accompanied by an unstained sample, if available. Information as to the manufacturer and type of paint should also be obtained. Tarnishing of silverware or electrical contacts are usual indications of the presence of sulphide gases in the air. Where any type of damage due to corrosion has occurred (aluminum sidings, automobiles, wire fences), it is best to have Laboratory staff inspect the damage and collect the sample for analysis. Gas detection tubes (Draeger, Kitagawa) can be used for a large number of gases. These can be purchased commercially. For sampling organic vapours, the Laboratory will supply specially prepared absorbent tubes or sampling bags. For gas damage to plant material, consult the Phytotoxicology Section (965-4516).

#### IV SAMPLE SUBMISSION

#### A - I GENERAL CONSIDERATIONS

Samplers must be aware of provincial, federal, and internatinal regulations controlling the transport of hazardous goods (samples), and act accordingly. In particular, samplers must adhere to the requirements of WHMIS (Workplace Hazardous Material Information System), a recent amendment to OHSA.

When submitting samples for analysis of organic contaminants, be as specific as possible about the types of compounds to be determined, and also, when a specific source of contamination is suspected, send samples of the source material for comparison. In all cases, the use of glass bottles with aluminum foil or Teflon-lined caps is a necessity for organic samples (see Table IV for details of sample containers).

It is difficult to sample foams. This may be best achieved by sampling just the foam with a pomade jar, then breaking the emulsion, and repeating the process until sufficient volume is obtained (usually at least 10 times). In most cases, it is more useful to collect samples of the originating liquid, rather than the foam itself, for chemical analysis.

Identification of unknown contaminants is very time-consuming. Samples should be as large as possible (within reason: take into account the suspected concentration of the contaminant) to allow a wide range of exploratory tests. Laboratory staff must be notified before submitting large quantities of hazardous or obnoxious wastes (i.e., mercaptans). The sampler should indicate whether qualitative or quantitative results are required. Any available information concerning the sampling point, possible contaminants, and industries implicated, is extremely important for such samples. See below regarding direct communication with the analyst. Organic compounds not readily identified by the other units in the Organic Sections, may be submitted to the Mass Spectrometry unit for analysis.

Samples which are not homogeneous present analytical difficulties because it is virtually impossible to take a representaive aliquot. If the sampler is only interested in one phase (aqueous, solid or immiscible organic), he should label the submission form with the appropriate test code. Otherwise, the laboratory will consider the whole sample, and take aliquots of the mixture.

Samples sent to the Central Laboratory, Toronto, may be analyzed by several sectional laboratories; the distribution of analyses among these sections is shown in Table 1. Sample processing is much more efficient if the sampler submits separate sample containers if testing is to take place in more than one of these sectional laboratories. Your cooperation in this respect will be appreciated by everyone involved, and it will be possible to obtain the results from some laboratories earlier than others, rather than having to wait for all tests to be completed.

Many samples are perishable. Some tests (i.e., BOD<sub>5</sub>) are performed on specific days of the week. The sampler is reminded that the delivery of samples to the Laboratory should coincide as closely as possible with the analytical schedule. Delivery on Friday afternoons or before holidays should be avoided.

#### A - II SAMPLE LABELLING AND COMMUNICATION WITH THE LABORATORY

Samples must be clearly labelled and contain the following information:

- a. A sample (sender's) number. The use of a simple field numbering system is encouraged. Please use a logical numbering system (such as downstream in a river).
- b. Some other identification, normally the sample source or type (e.g., "Lake Temagami Sharp Rock Inlet").
- c. Presence of any chemical preservative added; all others will be kept refrigerated or frozen (i.e., as received) if appropriate, until the time of analysis.
- d. When appropriate, indication of a single specific analysis required for that one sample bottle; i.e., when the sample has been preserved for resin and fatty acids analysis, it should be labelled "For Resin Acids", or when submitted for preconcentration and heavy metal analysis, it should be labelled "For Preconcentration".
- e. Samples which contain unusual or potentially dangerous substances, (arsenic, cyanide, mercury, etc.), must be labelled for the protection of Laboratory personnel. For example, an orange tape band around the sample container indicates that cyanide may be present. Also note any sample contents which may interfere with analyses. Many reactions which produce false analytical results can be reduced or eliminated if the analyst is forewarned.

## BOTTLE LABELLING IS MORE IMPORTANT THAN EVER BEFORE, SINCE IT IS OFTEN THE ONLY INFORMATION THE ANALYST SEES.

Direct communication with the analyst (Appendix I) may be achieved in two ways:

- a. Telephone call or visit to Laboratory Services.
- b. A note accompanying samples and addressed to the analyst in charge.

In all cases, a telephone call is recommended before submitting samples which need special attention. The sampler can then verify that the analysis is possible, find out who will be responsible, and when the results should be available. In addition, he may arrange a mutually agreeable time to submit the samples and meet at the laboratory with the analyst, or at least determine who should receive the note describing the samples. The note should be written on the bottom of the Submission Sheet and be marked "PLEASE NOTIFY (analyst's name) ON ARRIVAL". Sample reception will then contact the analyst as soon as the samples are unpacked.

#### A - III PARAMETERS AND TEST CODES

Parameters outlined in Table I must be specified as LIS test codes on the Request for Analysis sheets. Most users have lists of codes appropriate to their needs.

A test code must be specified accurately. Spelling a test code incorrectly may result in the performance of the wrong test. The test code does not specify which lab is to perform the test unless there is only one place where the test can be done.

Additional test code information can be obtained from your Branch representative on the LIS Users Committee or from the Client Services Hotline (416 235-5839).

#### A - IV PARAMETER GROUPINGS: GROUP CODES

Although the laboratories have analytical capabilities for many parameters, certain compatible groupings are requested frequently. Such group requests are usually associated with routine monitoring programs and/or specific projects. It is the nature of the groupings to allow analysis of all the specified parameters on a single or duplicate sample bottle.

Requests for any of these groupings should be made only when ALL the parameters are required. Otherwise, delete tests from the group by noting them in "Deletion From Group" box on Request for Analysis Sheets.

Unnecessary tests tax the analytical capacity of the laboratories.

Specific environmental problems usually require specific analyses to be performed and, therefore, use of these groups is of little value. Large projects and studies may find it advantageous to use a unique grouping and these may be established after consultation with the Laboratory Computer Systems Group, through the Client Services Hotline (416 235-5839).

#### A - V COMPLETING SUBMISSION FORMS

There are two basic documents used in submitting a sample to the Laboratory for analysis, namely:

- The <u>Submission</u> form (Figure III), which is used to enter data that is common to all samples in a submission such as sampling program, sampler's name, etc., and
- The Request for Analysis form (Figure IV), which is used to enter the sample information and the tests required.
   Data for several field samples may be entered on each sheet. If there are more samples than can be accommodated on one form, use as many sheets as required.

On the following pages, the individual fields on both forms are explained.

Provision has also been made to capture field results unique to a program. This data will be passed to the Sample Information System (SIS) for storage. Field results cover such items as water temperature, sample depth, etc., and are entered on forms prepared by the specific program chiefs.

#### 1. CODING REQUIREMENTS FOR SUBMISSION DOCUMENT

The following is to be used as a guide to filling in of the Submission Document.

Lab. Submission No. use only

The submission number is a unique number for any particular submission of samples and <u>must not</u> be duplicated or reused for any other submission. The submission number is usually pre-printed on the form in a sequential series for each Region or major sampling program. Alternatively, some Regions, Programs, or Branches have assigned a coded sequence of submission numbers to their users. If the submission number is <u>not</u> already on the form or if you are <u>not</u> using an assigned sequence, the submission number must remain blank, as sample reception staff will fill it in.

NOTE: THIS UNIQUE SUBMISSION NUMBER IS EXTREMELY USEFUL IN TRACKING THE ANALYSES THROUGH THE LABORATORY AND THEREFORE EVERY EFFORT SHOULD BE MADE BY THE SAMPLER TO RETAIN THIS NUMBER IN A SAFE PLACE SO THAT IT CAN BE GIVEN WHEN REQUESTING INFORMATION FROM THE LABORATORY.

From Field Sample No.

- Enter the first field sample number shown on the Request for Analysis or Field Data forms.

To Field Sample No.

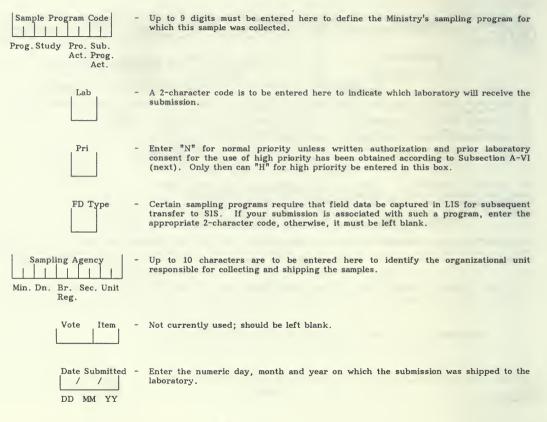
 Enter the last field sample number shown on the Request for Analysis or Field Data Forms.

Page of \_\_\_\_

Enter 1 in the first box and the <u>total</u> number of sheets in the submission, i.e., the number of Request for Analysis pages plus one.

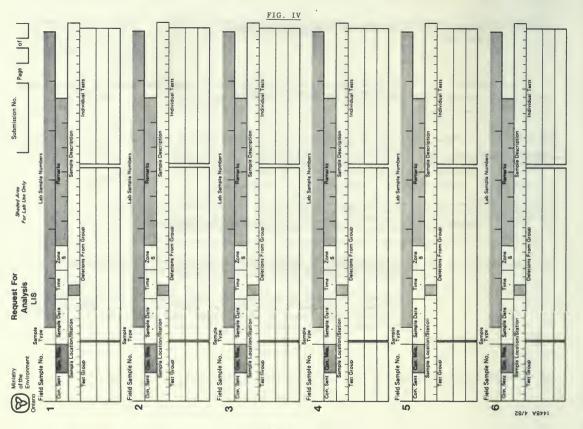
NOTE: The above 3 fields assist the Sample Reception staff in ensuring that all samples and sheets have arrived at the laboratory.

	Ministry of the Environment	Submission	Lab. use Only	No.	From Field Sample No To Field Sample No	Page of of
ntario	Project/ Mun. Code	Sample Program Code  Prog Study Pro Suc Act Prog Act Act Municipal/Project	Lab Pr. FD	Ma Do		Submitted / / / / / / / Phone
	Chent Code 1	Report to Name	P	one	Establishment	
		Address	1	City	Province (Country)	Postal Code
			Сорнез	To		
Client (	Code 2	Name	Phone	C en: Cooe 3	Name	Phone
		Establishment			Estab ishment	
		A did/ Res			Address	
	City	Province: Zountry	Posta Code	CTV	Province Country	Posta Code
Client	Cope 4	Name	Phone	C en: Code 5	4000	Prigne
		Estab shime in		-	Esrac shment	
		Address			Appress	
	City	Province Country	Posta Code	C ty	Province Country	Posta Code



	Project/ Mun. Code	- Not c	urrently used;	must be left blank.	
Municipa	al/Project			may be entered here to samples originate.	indicate the municipality or geographical
Name of Sampler	Phone	- Enter numb		surname and initial (up	to 20 characters in total) and telephone
Client Code	1 Report to	(Name)	Phone	Establishment	
	Address		City	Province(Country)	Postal Code

- Enter the client code and name of the primary recipient of the final report or, if this
  person doesn't have a client code, complete all other fields.
- Although the format of the areas for Client Codes 2-5 is different, the same information is required for each client who should receive a copy of the final report as for the primary client. If codes are used, other client identifier fields should be left blank.



#### 2. CODING REQUIREMENTS FOR REQUEST FOR ANALYSIS DOCUMENT

The following is to be used as a guide to filling in of the Request of Analysis Document.

Submission No.

- Transcribe the submission number from the Submission Sheet.

Page of

- Enter the page number of this sheet and the total number of pages in the submission. Since the Submission sheet is page 1, the first Request for Analysis sheet is page 2.

NOTE: A field sample is defined as the entire group of samples (in suitable containers and preserved appropriately for the tests requested) taken at a given sampling point at a given time. For each field sample, the following set of data items is required.

Field Sample No.

- Up to eight characters can be used to number field samples. Since this number also appears on the sample label, many users employ very simple numbering schemes. Use of a simple numbering scheme in a logical sequence will assist in the allocation of lab numbers by Sample Reception staff.

Sample Type  For some sampling programs, specific instructions have been given for field staff to enter the appropriate code.

However, in most cases, this area is filled in by Sample Reception staff. Unless you have received instructions to the contrary, leave this area blank.

Con. Sent

 Enter the total number of containers (suitable bottles, preserved appropriately) for this field sample.

Sample Date	Time	Zone 5
-------------	------	-----------

- Enter the date (in DD/MM/YY format) and, if appropriate for data interpretation, the time (24 hour clock) that the sample was taken. Time zone 5 (Eastern) is appropriate for the majority of the province. The western portion should use zone 6 (Central).

Sample Location/Station	-Enter the appropriate station identifier code, if utilized for the sampling program.
	Sample Location Description
	- Included on the newer Request for Analysis forms is this field of up to 40 characters for an optional alphanumeric description of the sample location code.
	Sample Description
	- Up to 32 characters can be used here to describe relevant features of the sample.

Test Group	Deletions from Group	Individual Tests

- These three columns are used jointly to specify the complete set of tests for all containers of this field sample.

NOTE: Tests have been given codes (up to 6 characters each) and many combinations of tests have been grouped to simplify the work of field staff as well as data entry personnel. The valid codes for tests and test groups are available from members of the LIS Users Committee (Table V) or from Laboratory staff. The total testing requirements of the field sample are specified by selection of the appropriate combination of test groups and individual tests.

Enter test group codes only in the lefthand column. If any of the tests included in the groups you have selected are not required, enter their individual test codes in the centre column. If individual tests are required, specify their codes in the righthand column.

Additional information, such as messages to analysts, should be written in the blank area at the bottom of the <a href="Submission">Submission</a> sheet. It will be photocopied and passed on to the appropriate Laboratory staff.

## TABLE V

## LIS USERS COMMITTEE

Air Resources Branch
Water Resources Branc
Central Region
West Central Region
Southeastern Region
Southwestern Region
Northeastern Region
Northwestern Region

Diane Green
Brian Whitehead
Dhan Sharma
Gus Perkons
Stan MacBeth
Walter Cook
Gerry Myslik
Pat Leung

(416)	965-1634		
(416)	323-4828		
(416)	424-3000		
(416)	521-7697	Hamilton	
(613)	549-4000	Kingston	
(519)	661-2266	London	
(705)	675-4501	Sudbury	
(705)	475-1275	Thunder	Bay

#### A - VI. HIGH PRIORITY SUBMISSION PROTOCOL

Samples are designated as "EMERGENCY" when collected as the result of a contingency which poses a direct threat to human health or which may lead to an emergency environmental situation.

Samples must be collected according to recommended laboratory procedures.

Each MOE Region and Branch must designate a senior staff member and alternate person who will co-ordinate high priority submissions for their specific area.

High priority submissions must be authorized by this senior staff member and receipt of high priority samples must be preceded by a phone call to either the Central Laboratory Emergency Response Co-ordinator or to any appropriate section Emergency Response Task Force (ERTF) representative or Manager (Table VI).

The laboratory personnel contacted will obtain a full description of the problem and sampling details and will ensure that the samples are received when and as described. The contact person is also responsible for ensuring that Sample Reception staff are notified and that the submission is assigned a "H" priority, and for contacting ERTF representatives in each affected laboratory section. An estimated completion date will be established and transmitted to the client.

All samples must be submitted through LIS with Submission and Request for Analysis forms completed and included with the samples, if possible, or forwarded as soon as possible. A <u>signed</u> authorization form from the senior regional co-ordinator must also accompany samples or be forwarded as soon as possible. Forms with photocopied signatures are not acceptable.

Submissions not meeting these requirements will be designated as Normal (N) priority and treated as such.

If arrangements have been made with ERTF personnel, results will be phoned-in to the client immediately upon completion of specific analyses prior to printing of a final LIS report.

The ERTF will meet every 2 to 3 weeks to review the status of all high priority submissions and to expedite completion of analyses within the estimated time limit.

If the laboratory cannot complete the analysis within the defined time limit, the ERTF representative of the section responsible for the delay must contact the client to explain the problem and assume responsibility for ensuring completion of the work by a mutually agreeable date.

## TABLE VI

## EMERGENCY RESPONSE TASK FORCE (ERTF) LABORATORY SERVICES BRANCH

(all area codes are 416)		Business
Central Co-ordinator	Rusty Moody	235~5863
ERTF REPRESENTATIVES		
Water Quality Inorganic Trace Contaminants Trace Organics Drinking Water Organics	Vera Turner Liz Pastorek Joe Osborne David Hall Vince Taguchi	235-5873 235-5855 235-5759 235-5910 235-5902
SECTION MANAGERS		
Water Quality Inorganic Trace Contaminants Trace Organics Drinking Water Organics	Joan Crowther George Kanert George Crawford Vacant	235-5868 235-5848 235-5757 235-5906
Senior Environmental Scientist (Organics)	Otto Meresz	235-5762
AFTER HOURS PHONE Spills Action Centre.		965-9619

## A - VII. SAMPLE CONTAINER REQUISITION AND SHIPPING PROCEDURES

Sample containers may be requisitioned according to need using the information provided in Table IV.

Certain projects or studies may require the use of <u>special container types</u>, and appropriate enquiry should be made prior to requisition.

Courier services provide the fastest and most reliable service for the shipment of environmental water samples in Ontario. Air express, parcel post, bus companies and other services discourage the shipment of water samples because of the damage caused to other shipments when breakage occurs.

Samplers must be aware of provincial, federal, and international regulations controlling the transport of hazardous goods (samples), and act accordingly. In particular, samplers must adhere to the requirements of WHMIS (Workplace Hazardous Material Information System), a recent amendment to OHSA.

NOTE: Contract numbers are important as they provide the only means for tracing a lost shipment. Every shipment is assigned a contract number, but it is generally up to the sampler to attach this contract number to each carton of his shipment. Identification stickers are provided by the courier companies upon request. Samplers are urged to keep a record of all their contract numbers.

## TABLE I ANALYTICAL TESTING CAPABILITIES - LABORATORY SERVICES BRANCH

Please consult Tables II, III, and IV for Sampling Requirements

W - Wat I - Inc	nking Water Orga er Quality Section rganic Trace Conf ace Organics (TO)	(WQS)		•	•		T -	London Reg Thunder Ba Kingston Re	y Regio	nal I	Labora		.у	
MAJO	OR IONS V	V I	L	Т	K		MI	ETALS	W	I		L	Т	K
Alkalinity	>	ζ.	Х	Х	X	Aluminum				х			Х	
Calcium	>	X	X	X	X	Antimony				X			X	
Chloride	>	X	X	X	X	Arsenic				X			X	
Conductivity	>	ζ	X	X	X	Barium				X			X	
Hardness	>	(	X	X	X	Beryllium				X			X	
Magnesium	>	X	X	X	X	Bismuth				X				
Potassium	2	XΣ	X	X	X	Boron				X			X	
Silicates - Reacti	ive >	(	X	Х		Cadmium				X			X	
Sodium	2	XΧ	X	X	X	Chromium -	Hexa	valent		X				
Sulphate	>	X	X	Х	X	Chromium				X			X	
•						Cobalt				X			X	
						Copper				X			X	
					_	Iron				X		X		X
						Lead				X			X	
						Lithium				X			X	
						Manganese				X			X	
				-	_	Mercury				X			X	
						Molybdenum				X			X	
						Nickel				X			X	
NUTI	RIENTS V	۷ I	L	Т	K	Selenium				X			X	
						Silver				X			X	
Ammonia Nitroge	n 2	XΣ	X	X	X	Strontium				X			X	
Nitrate Nitrogen		XΣ	X	X	X	Tellurium				X			X	
Nitrite Nitrogen		ζ	X	X	X	Thallium				X				
Nitrogen - Total	Kjeldahl 2	( X	X	X	X	Titanium				X			X	
Phosphorus - To		<b>Χ</b> Σ	X	X	X	Uranium				X				
Phosphorus - Re		ζ.	X	X	X	Vanadium				X			X	
						Zinc				X			X	

## TABLE I (Cont'd) - ANALYTICAL TESTING CAPABILITIES

## Please consult Table II for Sampling Requirements

ORGANIC	WI	PLTI		OTHER	WI	LTK
Base Neutrals/Acid Extractables		X	X	Acidity	X	XXX
				Algae *		
Ricebarias   Owners Depart (RODS)	X	XX:	v	Asbestos	X X	
Biochemical Oxygen Demand (BOD5) Carbon - Free (Elemental; Hivol)	X	XX.	X.	Carbonate (Hivol)		
Carbon Dioxide	X			Chlorine (Total Residual)	X	
Carbon - Dissolved Inorganic	X			Chlorine - Total (X-ray)	X	
Carbon - Dissolved Inorganic	X			Chlarophyll Colour - True	X X	XXX
Carbon - Inorganic	X	X		Colour Dilution		XXX
Carbon - Total (Hivol)	XX	X			X X	
Chemical Oxygen Demand (COD)	X	XXX		Corrosivity (Industrial Waste) Cyanide	X	
Dioxin	^	AAA	X	Dustfall	X	XX
Foams *		X	Α	Fluoridation Rate	X	AA
Hydrocarbon Gases		X		Flashpoint (Indust. Waste)	X	
Mass Spectrometric Services		Λ	Х	Fluoridation Rate	X	
Mercaptans - see Volatile		X	Λ	Fluoride	хх	XXX
Sulfurous Organics		Λ		Hydrogen (Indust. Waste, Soil)	X	АЛА
Methane - see Hydrocarbon gases		X		Leach test (Indust. Waste)	X	
Methylene Blue Active Substances	X	X		Loss on Ignition	X	XXX
Petroleum Hydrocarbons (Gasoline)	**	x .		Nitrogen (Indust. Waste, Soil)	X	AAA
Phenolics - Reactive	X	X		Oxygen - Dissolved	x	XXX
Polybrominated Biphenyls	21	X		Particle Size Analysis (Laser beam)	X	AAA
Polychlorinated Biphenyls		X	X	Particle Size by Microscopy (EM Unit)	X	
Polynuclear Aromatic Hydrocarbons		X	X	Particulate Identification (Complaint)*	X	
Purgeables		X	X	pH	XX	XXX
Resin and Fatty Acids		X		Phytoplankton*	****	20000
Solvent Extractables		X		Reactivity (Indust. Waste)	X	
Tannins and Lignins		X		Sieve Analysis	X	
Tracer Dyes		X		Solids - Dissolved	X	XXX
Trihalomethanes		X	X	Solids - Ignited	X	XXX
Vinyl Chloride		X		Solids - Suspended (by filtration)	X	XXX
Volatile Sulfurous Organics		X		Solids - Total	X	XXX
				Sulfation Rate		X
				Sulfide	X	X
				Sulfur - Total	X	
				Suspended Air Particulates - Total	X	X
*Aquatic Biology Section: Algal taxon	nomv. a	and		Turbidity	X	XXX

<sup>\*</sup>Aquatic Biology Section: Algal taxonomy, and examinations of phytoplankton, periphyton, zooplankton, slime, scum, foams, and biological nuisance organisms are performed at the Central Laboratory by the Aquatic Biology Section, Water Resources Branch.

## TABLE I (Cont'd) - ANALYTICAL TESTING CAPABILITIES

## Please consult Table II & Table III for Sampling Requirements

PESTICIDES			MICROBIOLOGY	CLTK
Pesticide analyses on aqueous effluent fish, other biota, soils, sediments, air precipitation samples are analyzed by Section; drinking, ground and surface associated with sewage or industrial el analyzed by Drinking Water Organics  Carbamate Insecticides/Herbicides Chlorinated Aromatics Chlorophenoxy Acid Herbicides Organochlorine Pesticides Organophosphorus Insecticides Phenyl Urea Herbicides Triazine Herbicides  NOTE: The Thunder Bay Regional La analyzes biota for organochlor	r cart Trace wat ffluer Section	ridges and e Organics er (not ats) are on.	C = Central Laboratory, Toronto L = London Laboratory, T = Thunder Bay Laboratory, K = Kingston Laboratory  Fecal Pollution Indicators Coliforms - Total Coliforms - Fecal Enterococci Escherichia coli Fecal Streptococci Presence Absence Procedure Pseudomonas aeruginosa Salmonella sp.  Industrial/Agricultural Pollution Indicators Klebsiella sp. Nitrogen cycle bacteria	XXXX XXXX XXX* XXXX XXXX XXXX XXXX
SEDIMENTS AND SOILS	I	T	- Denitrifying bacteria - Nitrobacter sp Nitrosomonas sp. Phenol Degraders Sulfate Reducers Sulfur Oxidizers	XXX XXX XXX X X XXXX
Hot Acid Extraction (Metals) Base Neutral Capacity P, TKN, C, CO <sub>3</sub> , S, Cl, F	X X X	Х	Nuisance Organisms	xxxx
Physical Loss on Ignition Moisture Content Particle Size Distribution	X X X		Organic Enrichment Indicators Fungi Heterotrophic bacteria - Surface Water - Treated Water	xx xxx xxx
			Taxonomy	XXX*

#### TABLE II - SPECIFIC PARAMETER INFORMATION

A number of the determinations listed in Table II may be applied to several sample matrices. The information in the table is for aqueous samples unless otherwise stated. Other eligible matrices are indicated by an initial in parentheses in the comments column, and sampling information is available from the appropriate laboratory section. Ambient air samples are collected in aluminized plastic bags, 5 to 22 L.

H= Hi-Vol filters V= Vegetation S = Sediments & Soils A = Ambient Air B= Biomaterials, Fish
D= Dustfall

P = Aquatic Plants

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common
MAJOR IONS				
Alkalinity	Glass or Plastic	None	75 mL	
Calcium	W	R	75 mL	
Chloride	w .	W	50 mL	
Conductivity	w .	W	75 mL	Specific Conductance
Hardness	W	Ħ	75 mL	•
Magnesium	#	W	75 mL	
Potassium	W		40 mL	
Silicates - Reactive	Plastic only	W	50 mL	Silica
Sodium	Glass or Plastic	W	40 mL	
Sulphate	н	W	50 mL	
NUTRIENTS				
Ammonia Nitrogen	Glass or Plastic	Refrigerate	75 mL	
Nitrate Nitrogen	(not linear polyethylene)	Ħ	W	
Nitrite Nitrogen	W	W	Ħ	
Nitrogen - Total Kjeldahl	l "	R	19	
Phosphorus - Total	W	W	W	
Phosphorus - Reactive	π	W	n	Soluble Phosphorus, Orthophosphate

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
METALS				
Aluminum	Plastic or Glass(2)	HNO <sub>3</sub> to pH of 2	100 mL, (500 mL	(H, V, S, D, B)(4)
Antimony	н	(approx. 20 drops	required for preconcentration	
Barium	Ħ	per litre)(1)	or ultra-trace analysis)	
Beryllium	- и	11	п	91
Cadmium	n	Ħ	TI .	91
Chromium	n	W	Ħ	11
Cobalt	п	91	π	99
Copper	я	Ħ	Ħ	n
Iron	W	W .	π	п
Lead	11	Ħ	H .	Ħ
Lithium	п	Ħ	n n	н
Manganese	11	н	Ħ	n
Molybdenum	н	п	Ħ	19
Nickel	Ħ	Ħ	Ħ	н
Silver	Ħ	Ħ	11	н
Strontium	n	Ħ	n	H
Tellurium	Ħ	н	11	-11
Thallium	Ħ	Ħ	Ħ	н
Titanium	π	Ħ	11	n
Uranium	Ħ	Ħ	H	н
Vanadium	m m	11	Ħ	п
Zinc	Ħ	11	Ħ	т
Arsenic	W.	None	50 mL	(V, S, D, B)
Boron	Plastic only	HNO3 as above	100 mL	(V, S, D, B)
Chromium (hex)	Glass only	None	100 mL	(V, S, D)
Mercury	Glass only	$HNO_3 + K_2Cr_2O_7(3)$	200 mL	(V, S, B)
Selenium	Plastic or Glass(2)	HNO <sub>3</sub> as above	100 mL	(H, V, S, D, B)

(1.) Nitric acid preservative should be added AFTER the sample is placed in the bottle. If the sample contains visible suspended solids or where a hazardous chemical reaction between the sample and the acid may occur, submit the sample unpreserved.

(2.) PET 500 containers are required for ultra-trace analysis; glass bottles, if used, must have non-metallic cap liners.

(3.) A special milk dilution bottle (stores #8c) is used for Hg samples. Add 1-2 mL HNO<sub>3</sub> per 250 mL, followed by at least 10 drops of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. The K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution should produce a definite yellow colour. Omit preservation where dangerous reactions may occur, or where the sample is heavily contaminated with organic material. If in doubt, contact Darryl Russell (235-5857).

(4.) Most of the metals listed can be determined also on Hi-Vol filters (H), vegetation/soils (V), sediment (SE), and dustfall (D); some of the metals listed can be determined on biomaterials (B). For specific metals, contact appropriate

I.T.C. staff (Appendix I).

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
ORGANIC				
Acid extractables	Glass (6)	Refrigerate	1 L	(7)
Base/neutral extractables	Glass (6)	Refrigerate	1 L	(7)
Biochemical Oxygen Demand	Glass or Plastic	Refrigerate	500 mL	BODs - Mark lid
Carbon - Free (Elemental)				with green tape (H, S)
Carbon - Dissolved Inorganic	Glass or Plastic	Refrigerate	50 mL	(11, 3)
Carbon - Dissolved Organic	Glass or Plastic	Refrigerate	50 mL	
Carbon - Inorganic	Glass or Plastic	Refrigerate	50 mL	(H, S)
Carbon - Total	Glass or Plastic	Refrigerate	50 mL	(H, S)
Chemical Oxygen Demand	Glass or Plastic	Refrigerate	25 mL	C.O.D.
Dioxin	Glass, Teflon-liner (6)	Refrigerate	1 L	-Consult DWO Section
Foams Freons	Glass Glass	Refrigerate		-Consult TO Section -Consult TO Section (A
G.C./M.S. Characterization; - Extractables - Purgeables	Glass, Teflon liner (6) Glass	Refrigerate Refrigerate	1 L (See Note 8)	-Consult M.S. Unit - <u>No</u> air space
Hexachlorobenzene	Glass, Teflon liner (6)	Refrigerate	(6)	нсв
Hydrocarbon Gases Mercaptans	Glass	Refrigerate	1 L	-Consult TO Section -see Volatile
Methane	-	-	-	Sulfurous Organic

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Parameter	Container	Preservation Technique	Minimum Volum Required	e Comment or Common Name
ORGANIC				
Petroleum Hydrocarbons(Gasoline) Phenolics - Reactive	) Glass Glass	Refrigerate H <sub>2</sub> SO <sub>4</sub>	1 L 250 mL	A special bottle containing preservative is available.
Polybrominated Biphenyls Polychlorinated Biphenyls	Glass, Teflon liner (6) Glass, Teflon liner (6)	Refrigerate Refrigerate	(6) (6)	PBB PCB (for drinking, surface & groundwater, use (OWOC) scan
Polynuclear Aromatic Hydrocarbon Purgeable Organics Resins and Fatty Acids	Glass Glass Glass	Refrigerate Refrigerate Refrigerate	1 L (See Note 8) 1 L	PAH No air space
Solvent Extractables Tannins and Lignins Tracer Dyes	Glass Glass Consult with TO Section	Refrigerate	1 L 200 mL	Ether Solubles
Trihalomethanes Vinyl Chloride Volatile Acids Volatile Sulfurous Organics	Glass Glass Glass Glass	Refrigerate	250 mL 1 L 25 mL 1 L	No preservatives (A) (combined, for sewage sludges) (A)

<sup>(6.)</sup> Special 1 L washed and baked containers are supplied (Pack #3 PCB). Fill container to shoulder, approximately 900 mL.

<sup>(7.)</sup> The same sample may be used for both acid and base neutral extractables.(8.) Volume depends on size of container (40 mL is sufficient volume with special glass vials that will soon be available).

TABLE II - SPECIFIC PARAMETER INFORMATION (Con 1'd)

Parameter	Container	Preservation Technique		um Volume Juired	Comment or Common Name
OTHER					
Acidity	Glass or Plastic	Refrigerate	50	mL	
Asbestos	Plastic	Refrigerate Analyze within 48 hours	s 1	L	(H, special filter)
Chlorine - Total Residual,	Glass	(9)	500	mL	-Consult Water Quality Section
Chlorophyll	Field filtration preferred	(10)	500	mL	
Colour - Apparent	Glass or Plastic	Refrigerate	75	mL	Hazen Colour Units
Colour Dilution	Glass or Plastic	Refrigerate	50	mL	
Cyanide Dustfall Fluoridation Rate	Glass or Plastic See text See text	NaOH to pH 11+	500	mL	
Fluoride	Glass or Plastic		50	mL	(H, V, B by ISE (11))
Loss on Ignition	Glass or Plastic	Refrigerate	500	mL	(D, H, S, V);
Oxygen - Dissolved	Glass	(9)	600	mL	Use BOD bottles
Particle Size Analysis Particle Size by Microscopy Particulate Identification (Complaint Samples)	Non-aqueous samp	only; Contact ITC ples only; Contact EM Uni Aquatic Biology Section, W		sources Br	ranch.

(9.) Due to the perishable nature of the measured constituents, analysis should ideally be performed on-site. For lab analysis, after proper sampling and refrigeration, samples must be submitted within 4 hours of collection with prior laboratory notification.

(10.) In the field, filter up to 1000 mL sample (minimum volume required 500 mL) through one nylon membrane filter (1.2 um pore size, 47 mm dia: Central Stores Cat. No. F036-1). Fold the filter in half avoiding exposure of the suspended matter, and place it on a filter pad (Cat. No. F066-1) in a plastic Petri dish equipped with a cover (Cat. No. D069-1). Record the volume (to the nearest 10 mL) of sample, which is filtered, on the Petri dish. If field filtration is not feasible, submit one litre of sample in a glass container. For either dishes or bottles, protect samples from the light, refrigerate, and ship as soon as possible.

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
OTHER				
рН	Glass or Plastic	Refrigerate	50 mL	
Sieve Analysis	-	-	-	Non-aqueous samples only
Solids - Filtered	Glass or Plastic	Refrigerate	75 mL	
Solids - Ignited	11	н	75 - 500 mL	See also Loss on Ignition
Solids - Suspended	11	Ħ	н	
Solids - Total	π	n	75 mL	
Sulfation Rate	-	-		See Section III.B.II.5
Sulfide	Glass	Zn acetate + Na <sub>2</sub> CO <sub>3</sub>	250 mL	Consult ITC Section
Sulfur - Total	_	(12)	-	prior to sampling
Suspended Air Particulates	-	-	-	TSP
Turbidity	Glass or Plastic		50 mL	

<sup>(11.)</sup> ISE = Ion Selective Electrode

<sup>(12.)2</sup> mL of 2N Zinc Acetate per litre, followed by dropwise addition of 5% Sodium Carbonate solution until precipitation complete.

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common
PESTICIDES				
Carbamate Insecticides/ Herbicides	Glass, Teflon liner (6)	Refrigerate	Fill container to shoulder approximately 900 mL	, Scan
Chlorinated Aromatics	n	11	и и	Scan
Chlorophenoxy Acid Herbicides	11	11	M .	Scan
Chlorophenols	Ħ	Ħ	W	Scan
Organochlorine Pesticides	Ħ	Ħ	Ħ	Scan
Organophosphorus Insecticides	N	11	W	Scan
Phenyl Urea Herbicides	n			Scan
Triazine Herbicides	n	н	n	Scan
AIR MONITORING				
Volatiles	Multi-phase absorbent	Refrigerate	3-36 L	VOC
Polychlorinated Biphenyls	Florisil	II STATE	IOm <sup>3</sup>	PCB
Chlorophenyls	Florisil	11	81	CP
Chlorobenzenes	Florisil	W	W	CB
Pesticides	Florisil	Ħ	n	
Polyaromatic Hydrocarbons	Glass fibre/XAD-2 resin	n	700-2800 m <sup>3</sup>	PAH
Organochlorine Pesticides	Glass fibre/XAD-2 resin	и	11	OC
Volatile Sulphur Compounds	Tedlar bag			

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
MICROBIOLOGY				
Fecal Pollution Indicators Routine				
Coliforms - Total	Sterile bottles containing sodium thiosulphate	Refrigerate	200	Membrane Filtration Incubation on Selective Agar
- Fecal	mosuiphate	et		19
Fecal Streptococci	11	er .	m m	11
Presence Absence Procedure	о п	et	m .	19
Pseudomonas aeruginosa Non-routine	11	61	Ħ	11
Enterococci	11	11	n	et .
*Escherichia coli	99	10	11	Ħ
*Salmonella sp.	11	п	500 - 1000	Membrane Filtration or Moore Swabs - Incubation In Selective Broth
Organic Enrichment Indicators *Fungi	71	n	150	Membrane Filtration Incubation on Selective Agar
Heterotrophic Bacteria - Surface Water	Ħ	er	11	Spot or Spread Plate Incubation on Non-Selective
- Treated Water	21	Ħ	**	Agar/ Membrane Filtration Incubation on Non-Selective
Nuisance Organisms	11	11	п	Agar/ Membrane Filtration Direct Microscopy or MPN Incubation in Selective Broth
Taxonomy	н	Ħ	11	Direct Microscopy and Biochemical Testing

 $<sup>\</sup>mbox{*}$  Only by special arrangement with the appropriate laboratory.

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Minimum Volume

Commont on Common

Parameter	Container	Technique	Required	Name
MICROBIOLOGY				
Specific (Industrial/Agricultur Pollution Indicators	al)			
*Klebsiella sp.	Sterile bottles containing sodium thiosulphate	Refrigerate  for chlorinated samples   (see text)	200	Membrane Filtration Incubation on Selective Agar
*Phenol degrading bacteria	я	*	18	MPN Incubation in Selective Broth
*Nitrogen Cycle Bacteria - Nitrosomonas sp Nitrobacter sp.	W	*	m m	*
- Denitrifying Bacteria *Sulphate Reducers	W N	Refrigerate only	n	п

# \*Sulphur Oxidizers Specialized Capabilities

Techniques for collection, handling and analysis of samples will be determined by project needs and must be decided by consultation—with the Microbiology Units. Methods are available to determine microbial biomass, metabolic activity transformation reactions, and to identify unknown bacterial isolates.

<sup>\*</sup> Only by prior arrangement with the appropriate laboratory.

TABLE II - SPECIFIC PARAMETER INFORMATION (Con t'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
AQUATIC BIOLOGY*				
Phytoplankton Biomass (Quantitative)	PET 500	Lugol's Iodine Solution - 2 mL/L	500 mL	Fill to capacity
Phytoplankton Biomass (Qualitative - Identification)	PET 500	Lugol's Iodine Solution and/or Refrigerate - 2 mL/L	500 mL	Preserved and/or live samples
Algae Identifications algal mass and/or slime accumulations	wide mouth container	Lugol's solution, Formaldehyde and/or Refrigerate - 2 mL/L	100 mL	Biological material should only be 1/10 of container volume topped with water
Nuisance Organisms algae, zooplankton, aquatic weeds, pine pollen, shoreline accumulations	wide mouth container	Lugol's solution, Formaldehyde and/or Refrigerate - 2 mL/L	100 mL	Biological material should only be 1/10 of container volume topped with water

<sup>\*</sup> Water Resources Branch

TABLE III(A)
TESTS PERFORMED BY THE SEDIMENT AND SOILS UNIT (ITC)

Type of Test	Sample S Minimum	ize (g) Optimum	Extracting Reagent	Parameters	Notes
Hot Acid Extraction	0.2	2 2	HCl/HNO3	Most metals	AAS
(Routine)	0.2	2	H2SO4/HNO3	Ag, Ti	ICP
	0.1	1	H <sub>2</sub> SO <sub>4</sub> persulphate	N, P	
Mild Acid Extraction	0.5	2	HCI O.	F	ISE
	0.1	1	HCl	CO <sub>2</sub>	.02
Water Extraction	3.0	10	Distilled water	Cl	IC
Particle Size Distribution:					
(a) Small Size	1.0	2.0			Based on dry weigh
(b) Large Size	50	100			Based on dry weigh
Loss of Ignition	2	5			
Moisture Content	5	10			
Combustion - Oxidizing	0.5	2		S, C	LECO

NOTE: For non-routine tests, it is advisable to consult with the Sediment and Soil Unit, 235-5855.

TABLE III(B)
TESTS PERFORMED BY INDUSTRIAL/DOMESTIC WASTE UNIT (ITC)

Type of Test	Sample Size (g) (Minimum)	Extracting Reagent	Parameters	Notes
Leach	500 g	0.5 N Acetic acid	Reg 309, Schedule 4	ICP analysis Colourimetric: anions
Corrosivity	1 L	Sample itself	Corrosivity	pH may be used
Flash Point	50 mL	None	Flash Point	
Calorific Value	50 g/50 mL	None	Heat BTU	
Reactive CN	100 g/100 mL	HCl/or H <sub>2</sub> SO <sub>4</sub>	CN	For listing/delisting Hazardous Wastes
Reactive H <sub>2</sub> S	100 g/100 mL	HCl/or H <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> S	For listing/delisting Hazardous Wastes
Acid Extraction	100 mL/50 g (sewage) 100 mL (landfill leachate) 100 mL (liquid ind. waste) 25 g (solid ind. waste)	aqua regia	Metals	ICP analysis

TABLE IV
SAMPLING SUPPLIES - CENTRAL STORES

## SAMPLING CONTAINERS

Type of Sample/Test	Container	Pack Contents	Pack Number	Special Features
Water, liquid wastes - general purpose	500 mL PET plastic	6 bottles	PET500-6	WM, PP cap
Water, Public Health domestic inspections	200 mL PP plastic	4 bottles	PACK=30	Includes sleeves
Sludges, sediments: - general purpose (including metals)	500 mL PET	6 bottles	PET500-6	WM, PP cap
- organics	250 mL WM brown glass	12 jars	PACK #5 PCB	Foil liner -containers baked
Routine Organic Extractables: (includes dioxin, pesticides, PCBs)	1 L brown glass	6 bottles	PACK#3PCB	Teflon liner
Aqueous Organic Identification	1 L clear glass	6 bottles	PACK#3	Foil cap
PCB in oil	30 mL glass	24 vials	PACK#21	PP cap
Phenols	250 mL glass (green label)	4 bottles	PACK#7S	Foil cap, with preservative
General chemical tests (mercury, sulphide)	250 mL glass (black label)	4 bottles	PACK#7C	Unsterilized, recycled

## TABLE IV (Cont'd)

## SAMPLING SUPPLIES - CENTRAL STORES

#### SAMPLING CONTAINERS

Type of Sample/Test	Container	Pack Contents	Pack Number	Special Features
Purgeables, trihalomethanes	250 mL glass (black label)	4 bottles	PACK#7C	Foil liner, unsterilized, recycled
Metals, precipitation	500 mL PET plastic	6 bottles	PET500-6	WM, PP cap
Soils analysis - general	125 mL glass	24 jars	PACK#S125	PP cap
Bacterial analysis	250 mL glass (red label)	4 bottles	PACK#7T PACK#7TF	Sterile bottles, thiosulphate added -Same, with foam liner
Aquatic Plants:  - Lake, river, raw water, wells  - Shoreline accumulations and scums	500 mL PET plastic, plastic bag	6 bottles	PET500-6	WM, PP cap

Glossary: WM - wide mouth

PP - polypropylene PET- polyethylene terephthalate

#### APPENDIX I

## **ENQUIRIES**

Enquiries regarding sampling and test procedures and the status of outstanding samples should be directed initially to the Client Services Hotline (416-235-5839). If necessary, you will be put in contact with the appropriate technical individual. A list of technical contact staff is provided below.

All samples received by the Laboratory Branch are assigned alpha-numeric codes according to sample type. As the analyses are completed, the results are entered into the computer (LIS system). When data for all tests have been entered, final reports are checked by senior staff and mailed. All original submission sheets are retained in the sample reception files for 18 months, after which they are destroyed. Laboratory staff are prepared to answer questions regarding the receipt and progress of samples but require the following information:

- a) Submission number and date of submission
- b) Name of sampling area as given on submission
- c) Field sample number and sample description
- d) Type of sample (e.g. water, river, sewage, industrial wastes, Great Lakes, algae, etc.)

NOTE: The submission number is the most important piece of information in tracking work through the Laboratory Information System. Please retain this record in a safe place and use it when requesting information as to the progress of your analytical work.

## WATER QUALITY SECTION Telephone - (416) 235-5870

Manager \*ERTF Representative Joan Crowther Vera Turner (416) 235-5868 (416) 235-5873

The Section is responsible for the analysis of all major ions, nutrients and physical properties in water (including precipitation, surface water, groundwater, sewage and industrial wastes). The section is also responsible for environmental microbiology tests.

General inquiries regarding results should be directed to the following staff based on sample classification.

## Classification Name

#### Chemical:

- -Chlorophyll
- -Great Lakes
- -Public Health Inspection
- -Industrial Waste
- -Precipitation
- -River (surface water)
- -Sewage
- -Water (Ground/Domestic)
- -APIOS Air Filters
- -Stemflow
- -Soil Leach ARB (APIOS)

## Microbiology:

- -Drinking Water
- -Lakes, Rivers and Wastes
- -Nuisance Organisms

Contact Staff (416) 235-5870

Ben Cheung Kathleen Hansen Maria Zanette Peter Campbell Bernard Wright Kathleen Hansen Ben Cheung Maria Zanette Bernard Wright Bernard Wright Kathleen Hansen

(416) 235-5870 Jim Clark/Steve Debreceni Michael Young/Susan Janhurst Michael Young/Garry Horsnell

\*ERTF =Emergency Response Task Force

## INORGANIC TRACE CONTAMINANTS Telephone - (416) 235-5845

ManagerGeorge Kanert(416) 235-5848Emergency Response RepresentativeLiz Pastorek(416) 235-5855

The ITC Section provides analytical services which include the determination of elemental and mineral constituents in a variety of sample types such as water, soils, vegetation, air filters, fish, etc.

General inquiries regarding results should be directed to the following staff based on sample classification.

Classification Name	Contact Staff	
Biomaterials Fish	Ram Sadana Ram Sadana	(416) 235-5861 (416) 235-5861
Metals in Water Fluoride Candles Hi-Vol Filters	D. Boomer J. Hipfner	(416) 235-5858 (416) 235-5856
Anderson Filters Dustfalls Hi-Vol Particulates	n n	27 20 20
Dichotomous Metals Industrial Wastes	" Jim Pimenta/George Wood	(416) 235-5854
Landfill Leachates Sediments Sewage Sludge	" Liz Pastorek Jim Pimenta	(416) 235-5855 (416) 235-5854
Precipitation, Stemflow Vegetation/Soil Court Cases	D. Boomer Liz Pastorek Rusty Moody	(416) 235-5858 (416) 235-5855 (416) 235-5863
Particulate Identification/Complaint Electron Microscopy, Radiochemicals, Legal	Rusty Moody Rusty Moody	(416) 235-5863 (416) 235-5863

### TRACE ORGANICS SECTION Telephone - (416) 235-5758

Manager ERTF Representative George Crawford Joe Osborne (416) 235-5757

(416) 235-5759

The Section is divided into four main functional units:

The current analytical capabilities of each are:

1. Biomaterials and Sediment/Vegetation Unit;

Currently the unit has capabilities for a broad range of extractable pesticides, herbicides, chlorophenols, PCBs, chlorinated benzenes, toluenes and higher molecular weight chlorinated industrial organics, and a limited range of other industrials such as phosphate ester plasticizers for hydraulic fluids.

## 2. Atmospheric Monitoring Unit:

Currently the Atmospheric Monitoring unit has capabilities for sampling and analysis for a range of pesticides, PCBs, polyaromatic hydrocarbons, and chlorinated industrial chemicals in the vapor and particulate phase in ambient air and stack emissions. The capability for analysis of organic vapors (both chlorinated and non-chlorinated compounds) in ambient air by absorbent trap-thermal desorption is available.

The unit has the capability for limited non-automated analysis of gas samples for gaseous hydrocarbons, sulphur gases, and chlorinated hydrocarbons.

## 3. Hazardous Waste and Mobile Operations Unit:

This unit has three functions: (a) co-ordination of all LSB regional mobile landfill investigations and/or on site monitoring studies, and determining related organic constituents; (b) the analysis of solids and hazardous/high concentration liquid wastes for toxic organics; and (c) characterization of solid and liquid wastes for investigations and litigation.

## 4. Wastewater Unit:

This unit provides analytical services for the measurement of trace organics in municipal and industrial effluents and leachates.

Based on the above criteria, general inquiries regarding trace organic results should be directed to the following staff based on sample classification.

## TRACE ORGANICS SECTION Telephone - (416) 235-5758

## Classification Name

Sediments/Soils

Landfill (any)

Water (any)

XAD resins

Trade Wastes

Mobile Operations

Biomaterials (not fish) Biomaterials (fish) Vegetation Air

Organic characterization/identification\*

Contact Staff

(416) 235-5758

Dan Toner

.

н

Brian Foster Yvonne Jones/Joe Osborne Yvonne Jones

Joe Osborne Brian Foster Joe Osborne

- 11

PCB's

<sup>\*</sup>Includes solvent extractables and petroleum hydrocarbons.

## DRINKING WATER ORGANICS SECTION Telephone - (416) 235-5900

Manager ERTF Representatives	Vacant David Hall Vince Taguchi	(416) 235-5906 (416) 235-5910 (416) 235-5902
	vince ragueni	(416) 233-3302

General inquiries, should be directed to the following staff based on parameter:

Parameter	Matrix	Scan	Contact Staff	
Polychlorinated dibenzodioxin and Polychlorinated dibenzofurans	water, fish/biota incinerator emissions soils/sediments special projects	PEDIOX	T. Thompson	(416) 235-5892
PCB's Chlorinated aromatics Organochlorine pesticides Organophosphate pesticides Chlorinated phenols Phenoxy herbicides Carbamates Triazines Phenylureas Trihalomethanes Volatile/purgeable organics Base/neutral/acid EPA extractables	- drinking, ground and surface waters	OWOC " PEOP OWCP " PECAR PETRI PEURH OCOHVI OPOPUP (OPOPRO) OPOBNO OPOAXO	D. Hall O. Hall O. Hall O. Wall O.W. Berg O.W. Berg O.W. Berg	(416) 235-5910 (416) 235-5907
Parameter Class Mass Spectrometry, GC/MS GC/MS Volatiles Extractables Speciated Phenols Special PAH's		PBVOL PBEXT OAPHNX OAPAHX	V. Taguchi V. Taguchi D. Hall D. Hall	(416) 235-5902 (416) 235-5910

AQUATIC BIOLOGY SECTION (WATER RESOURCES BRANCH)	Contact Staff	
Aquatic Plant Unit: Phytoplankton, Zooplankton, Periphyton Aquatic Toxicity Unit: Bioassessment Unit - general - sportfish program Biohazard Unit:	Gord Hopkins Ken Nicholls Gary Westlake Wolfgang Scheider Al Johnson Dave Rokosh	(416) 235-5811 (416) 235-5810 (416) 235-5797 (416) 323-4925 (416) 323-4927 (416) 235-5787
LABORATORY COMPUTER SYSTEMS - QA/QC SECTION		
Manager LIS/SIS Coordinator Quality Assurance/Quality Control	Larry Vlassoff Larry Vlassoff Don King	(416) 235-5805 (416) 235-5805 (416) 235-5838
SHIPPING AND RECEIVING		
Central Stores Sample Bottle Supply	Walter Wright	(416) 235-5741 (416) 235-5739
LONDON REGIONAL LABORATORY		
Chief Laboratory Services Chemistry Microbiology	Walter Cook Roger Rioux Gary Palmateer	(519) 661-2266 (519) 661-2240 (519) 661-2268
THUNDER BAY REGIONAL LABORATORY		
Manager, Utilities and Special Projects Chemistry, Trace Contaminants Chemistry, Water Quality Microbiology/Administration	Jim Stasiuk Patrick Leung Shirley Remmen Alice Chony	(807) 475-1275 (807) 475-1275 (807) 475-1275 (807) 475-1275

## KINGSTON REGIONAL LABORATORY

Chief Laboratory Services Chemistry Microbiology QA/QC Scientist

## Contact Staff

Stan MacBeth (613) 549-4000 Dave Ferguson " Art Ley/Neil Rickey " Vicki Kamphuis "

#### APPENDIX II

The following addresses should be used when shipping samples to the various laboratories:

### a) CENTRAL REGION - MAIN TORONTO LABORATORY

Ontario Ministry of the Environment, Central Stores, 125 Resources Road, Highway 401 and Islington Ave., Toronto, Ontario. M9W 5LI

# Mailing Address:

Ontario Ministry of the Environment Central Stores P.O. Box 213 Rexdale, Ontario M9W 5L1

#### b) SOUTHWESTERN REGION - LONDON LABORATORY

Ontario Ministry of the Environment, Southwestern Regional Laboratory, 985 Adelaide Street South, London, Ontario. N6E 1V3

## c) NORTHWESTERN REGION - THUNDER BAY LABORATORY

Ontario Ministry of the Environment, Thunder Bay Regional Laboratory, 421 James Street South, Thunder Bay, Ontario. P7E 2V6

#### d) SOUTHEASTERN REGION - KINGSTON LABORATORY

Ontario Ministry of the Environment, Southeastern Regional Laboratory, 133 Dalton Avenue, Kingston, Ontario KTL 4X6

Further enquiries regarding container requisitions, shipping, etc., should be directed to Central Stores in Toronto (Telephone 235-5739).













